DRAFT
Quality Assurance Project Plan Addendum

Time Critical Removal Action for the Refuse Area at the Georgia Pacific Corporation Kalamazoo Mill Property and the Former Hawthorne Mill Property

Allied Paper Inc./Portage Creek/Kalamazoo River Superfund Site
Kalamazoo, Michigan

June 2006

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
[1]Always add acid to water to prevent violent reactions.			
[2]Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect, or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix: the substrate of a test sample.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a <u>range</u> where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a pure substance with properties that mimic the analyte of interest but that is unlikely to be found in environmental samples.

Test Method: defined technical procedure for performing a test.

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TITLE: **Wet Chemistry**
Paint Filter

Updated by:	Signature:	Date:
Diane L. Harper Section Manager, Wet Chemistry	Diane L. Harper	1/24/06

Approved by:	Signature:	Date:
Diane L. Harper Section Manager, Wet Chemistry	Diane L. Harper	1/24/06
David W. Mazur Env. Health & Safety Coor.	DWMazur	1/24/06
Terese A. Preston Quality Manager	Terese A. Preston	1/26/06

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COPY # : Uncontrolled

ISSUED TO :

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with STL's QA Standard Practice Records

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) is used to determine the presence of free liquid in a representative sample of waste. This SOP was written using SW-846, 3rd Ed., Method 9095A as a reference. This method is applicable to any waste material and is used to determine compliance with 40 CFR Part 264.314 and 265.314.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable.

1.1.2 Reporting Limits

0 mL Free Liquid per 100 grams

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM).

1.2 Summary of Method

A weighed sample is placed into a supported paint filter. Any liquid that drops from the filter in 5 minutes is considered free liquid.

2.0 INTERFERENCES

The method is not subject to interferences, but some samples may damage the paint filter itself. Samples must not be frozen.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 Specific Safety Concerns or Requirements

No hazards exist in this test, other than the potential of the sample themselves. All samples should be considered toxic unless known otherwise.

3.2 Primary Materials Used

There are no materials used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

4.0 Equipment and Supplies

- fine mesh paint filter, mesh #60 (available from a paint store)
- glass funnel or paint filter support
- 100 mL glass graduated cylinder that is completely dry
- ring stand
- balance, top loading

5.0 Reagents and Standards

None.

6.0 Calibration (Non-Daily)

None.

7.0 Procedure

7.1 Quality Control Checks

A matrix duplicate (MD) is performed with each set of 20 or few samples.

7.2 Sample Preservation and Storage

Samples are stored at 4 $\pm$ 2°C prior to analysis; and are analyzed within 28 days of sampling.

7.3 Sample Preparation

Not Applicable.

7.4 Calibration / Standardization

The top-loading balance must have been verified on the day of use using class S weights per STL Chicago SOP # UQA-003, Balance Calibration.

7.5 Preventive Maintenance

Not Applicable.

7.6 Sample Analysis

7.6.1 Place a completely dry 100 mL graduated cylinder on a top-loading balance. Place a funnel in the graduate and a paint filter in the funnel. Tare the balance.

7.6.2 Add approximately, but not less than, 100 grams of sample to the paint filter. If it is not possible to obtain a sufficiently representative sample of 100 g, the analyst may use a larger sample size in multiples of 100 mL or 100 g. However, when larger samples are used, divide the sample into 100 mL or 100 g portions and test each portion separately. If any portion contains free liquid, the entire sample is considered to have free liquids. If less than 100 grams of sample is available for analysis, the section manager should be consulted.

Odd-shaped samples that do not conform to the filter shape should be reduced by cutting or light crushing to small pieces of less than 1 cm, taking care to include all aspects of the sample representatively. Do not grind the sample.

7.6.3 Let the sample drain for five minutes.

7.6.4 If any liquid collects in the graduated cylinder, the volume collected is reported as mLs/100 grams, and as a "fail". If no liquid collects, the result is reported as 0.0 mLs/100 grams and as a "pass".

7.7 Documentation

7.7.1 Analysis Logbook

The analysis of samples and standards is documented within the instrument run log which must be completed for each day's analysis. It is good practice to record a brief description of the sample when doing this test, such as "wet, black sediment' or "dry clay".

7.7.2 Reporting Results

Without rounding, enter the raw data into LIMS. The data book and LIMS must be 1st reviewed by the analyst and 2nd reviewed by a trained reviewer.

8.0 QUALITY CONTROL

8.1 QC Summary

The matrix duplicate (MD) must be within $\pm$ 20 RPD for the paint filter test.

8.2 Corrective Actions

Poor duplicates can result for non-representative 100 g sub-samples. Care should be taken to acquire the most representative sample aliquot possible.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Record the amount of free liquid collected in a graduated cylinder as 'Volume of liquid collected (mLs)/100 (grams or mLs)'.

9.2 If more than 100 grams of sample were tested in 100-gram aliquots, divide the total volume of liquid collected by the number of 100-gram aliquots to report as mL/100g. Results may also be entered as pass or fail, with any amount of free liquid a fail.

10.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Alkaline sample waste generated by the analysis will be collect in approved containers and poured into the Carboy labeled "Corrosive Liquid" waste using a funnel to reduce splashing.
- Acidic sample waste generated by the analysis will be collect in approved containers and poured into the Carboy labeled "Corrosive Liquid" waste using a funnel to reduce splashing.
- The soiled paint filter and solid residue will be collected in the white solid waste containers.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 9.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Attachment 1: Analysis Logbook/LIMS Forms

Historical File:		
	Revision 00: 08/29/90	Revision 04: 09/28/00
	Revision 01: 07/30/93	Revision 05: 09/23/03
	Revision 02: 02/11/98	Revision 06: 02/01/05
	Revision 03: 06/16/99	Revision 07: 01/23/06

Reason for Change: Revision 07:
- Annual Review
- Remove Labnet-specific references

U:\QC\SOP\WC\9095.doc

Attachment 1.

Example: Analysis Logbook w/ LIMS Forms

STL Chicago

Miscellaneous (IMA) Parameter Listing

Parameter	Analytical Method Number(s)	Hold Time	Reporting Limit	Units
Settable Solids	E 160.5	7 days	0.5	ml / L
Density / Specific Gravity	SM 2710F; D5057	None	0.1	g/cc; N/A
Physical Description		None	NA	NA
Oxidizer Screen				
Residual Chlorine	E 330.4; SM 4500Cl F	Immediate	0.2	mg/L
Langelier Index / Corr.	SM2330A+B	None	NA	NA
Ferrous Iron	SM 3500FeD	Immediate	0.05; 5.0	mg/L; mg/Kg

Miscellaneous

Miscellaneous (IMB) Parameter Listing

Parameter	Analytical Method Number(s)	Hold Time	Reporting Limit	Units
Flashpoint (Ignitability)	S1010 (Closed Cup)	None	200	deg. F
Flashpoint	ASTM D92 (Open Cup)	None	200	deg. F
Paintfilter	S 9095	None	0	mL/100g
Specific Conductance	E 120.1; SM 2510B	28 days	1	uhmos/cm

Miscellaneous

Note: Holding Times listed as 'None' default to 28 days and those listed as 'Immediate' default to 6 hours in the LIMS system.

Updated: 09/26/05

STL Chicago

Miscellaneous (IMB)

Parameter: ____________

LabNet Test Method: ____________ LabNet Batch #: ____________

Reporting Limit: ________

Drinking Water Set (Circle): YES NO

Prep Time: ________ Start ________ End

Analysis Time: ________ Start ________ End

Page #: ____________

Book #: ____________

Instrument: ____________

Conditions: ____________

Calculation: ____________

Stds. / QC / Sample #		Results	Units	% Rec RPD

Standard Traceability:

Reagent Traceability:

Eppendorf Traceability:

Note: Working Standards are prepared daily from the noted Stock Solutions.

Analyst Signature: ____________ Date: ____________

Reviewer Signature: ____________ Date: ____________

CHI-22-12-006/D-3/99

*** RUN LOG *** (V2)

1/27/06 7:16

Paint Filter Test Method Code..: 9095 Batch Code...: 171031	Status.......: RVWD Batch Date...: 01/23/06 Batch Time...: 955	User Name......: jmk QC Code........: STD Calc Code......:	Location Code..: 57222 Equipment Code.: Import Code....:

			TEST CODE	PAINTF	PFTEXT
			TEST POS	1	2
SAMPLE: Grp Pos	Sample ID	Dilution	Date / Time		
1 1	243608_17_S___		1/23/06 0740		0
1 2	243608_17_S_MD__1		1/23/06 0755	0	
1 3	243608_16_S___		1/23/06 0810		0

*** RAW TEST DATA *** (V2)

Paint Filter Test

Report Date: 1/27/06 7:16

Method Code..: 9095	Batch Date...: 01/23/06	QC Code........: STD	Equipment Code.:
Batch Code...: 171031	Batch Time...: 955	Calc Code......:	Import Code....:
Status.......: RVWD	User Name....: jmk	Location Code..: 57222	

SAMPLE:	Grp	Pos	Sample ID	Dilution	PAINTF mL/100g	PFTEXT Text			
	1	1	243608_17_S___		0	pass			
	1	2	243608_17_S_MD__1		0	pass			
	1	3	243608_16_S___		0	pass			

*** QC Summary *** (V2)

Paint Filter Test

Report Date: 1/27/06 7:16

Method Code..: 9095	Batch Date...: 01/23/06	QC Code........: STD	Equipment Code.:
Batch Code...: 171031	Batch Time...: 955	Calc Code.......:	Import Code....:
Status.......: RVWD	User Name....: jmk	Location Code..: 57222	

Grp	Smp	Sample ID	Pos	Test	Result	Known	Original	Alternate	QC Res	F	QC Res	F
1	2	243608_17_S_MD__1	1	PAINTF	0		0					

SEVERN TRENT STL

STANDARD OPERATING PROCEDURE
TOXICITY CHARACTERISTIC LEACHING PROCDURE
SW-846 Method 1311 / Non-Volatile TCLP
Applicable Matrices: Liquid, Solid & Multiphasic Waste

APPROVAL SIGNATURES

Laboratory Director:	Michael F. Wheeler, Ph.D.	Date: 5/13/05
QA Manager:	Kirstin L. McCracken	Date: 5/13/05
Department Manager:	Jon K. Zygmuntowicz	Date: 5/13/05

Proprietary Information Statement:

1.0 SCOPE AND APPLICATION

1.1. This SOP describes the laboratory procedure for the preparation of non-volatile TCLP extracts.

2.0 SUMMARY OF METHOD

2.1. Liquid wastes (<0.5% dry solid material) are filtered through a 0.6 to 0.8 um glass fiber filter and the filtrate is defined as the TCLP extract.

2.2. For wastes with $\geq$ 0.5% solids, the liquid (initial liquid phase), if any, is separated from the solid phase and stored for later analysis, if necessary, the particle size of the solid phase is reduced, and the solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 um glass fiber filter. If compatible, the initial liquid phase of the waste is added to the liquid extract, and these are prepared and analyzed together. If incompatible (multi-phase), the liquid extracts are not combined; they are prepared and analyzed separately with results mathematically combined to yield a volume-weighted average concentration.

2.3. This procedure is based on SW-846 Method 1311 Toxicity Characteristic Leaching Procedure, Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, Rev 1, December 1996.

3.0 DEFINITIONS

3.1. Percent Solids: fraction of a waste sample from which no liquid may be forced out by an applied pressure.

3.2. Spike: a known amount of an analyte added to a blank, sample or sub-sample.

4.0 INTERFERENCES

4.1. Potential interferences that may be encountered during analysis are described in the analytical SOP for the determinative method.

5.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.1 Specific Safety Concerns or Requirements

Protective clothing, safety gloves and eye shields should always be worn when performing this procedure.

5.2 Primary Materials Used

Table 1, Section 17.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

Agitation apparatus: Capable of rotating the extraction vessel in an end-over-end fashion at 30 rpm (±2 rpm).

Bottle Extraction Vessel: 2 L Amber borosilicate glass containers with sufficient capacity to hold the sample and the volume of extraction fluid needed. Polyethylene bottles may be used for the leaching procedure if the extracts are for inorganic analysis only.

Filtration Devices:

Filter Apparatus: A stainless steel filter apparatus lined with Teflon that employs positive pressure to achieve phase separation. The unit is pressurized using nitrogen with the vacuum gradually applied to approximately 1-10 psi, unless no liquid passes through the filter. The pressure is then gradually increased to a maximum of 40 psi. The internal volume capacity of the unit used is 1.5 Liter.

Filters: 0.7 um pore density acid washed filter

pH Meter

Top Loading Balance

Beaker or Erlenmeyer flask, glass, 500 mL.

Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

Magnetic Stir Bar

Thermometer; range of –20°C to 110°C

9.5 mm sieve

7.0 REAGENTS AND STANDARDS

Reagent Water. ASTM Type II water.

Hydrochloric acid (1N), J. T. Baker, HCl, reagent grade.

Nitric acid (1N), J.T. Baker, HNO_3, reagent grade.

Sodium hydroxide (1N), J. T. Baker, NaOH, Pellet, reagent grade.

Glacial acetic acid, J. T. Baker, CH_3CH_2OOH, reagent grade.

Extraction Fluid

Extraction fluid #1: Add 5.7 mL glacial CH_3CH_2OOH to 500 mL of reagent water (See Section 7.1), add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. The pH of this fluid should be 4.93 $\pm$ 0.05. Prepare as needed or every six months.

Extraction fluid #2: Dilute 5.7 mL glacial CH_3CH_2OOH with reagent water (See Section 7.1) to a volume of 1 liter. The pH of this fluid should be 2.88 $\pm$ 0.05. Prepare as needed or every six months.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

The requirements for sample collection should be described in the client's sampling plan. Preservatives should not be added to samples before extraction. Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 11.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. Acceptable sample and extract holding times are given in Table 2.

Table 2: Sample Maximum Holding Times (Days)

	From: Field Collection To: TCLP Extraction	From: TCLP Extraction To: Preparative Extraction/Digestion	From: Preparative Extraction or Digestion To: Determinative Analysis	Total Elapsed Time
Semi-Volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals	180	NA	180	360

9.0 QUALITY CONTROL

A blank consisting of the same extraction fluid as used for the samples must be extracted with every extraction batch.

At least one matrix spike must be analyzed for each analytical batch in order to determine whether matrix interferences exist..

The matrix spike solution for inorganic analysis is added after filtration of the TCLP extract and before preservation and should not be added prior to TCLP extraction of the sample.

In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

Additional QC required is described in the appropriate extraction and analytical SOP for the determinative method to be employed.

10.0 CALIBRATION AND STANDARDIZATION

Calibrate the analytical balance each day of use, prior to use.

Calibrate the pH meter each day of use, prior to use.

11.0 PROCEDURE

<u>Preliminary Evaluations</u>

Use a 100 g aliquot of waste to perform preliminary evaluations to determine percent solids and significance of percent solids, if particle size reduction is needed, and to determine which extraction fluid should be used for the TCLP extraction of the waste.

Percent Solids Determination

If the waste will yield no liquid (100% solid) when subjected to pressure filtration proceed to section 11.1.2 to determine if particle size reduction is needed.

If the sample is liquid or multiphasic, separate by filtration the liquid from the solid:

1) Pre-weigh the filter and the container (1 L amber) that will receive the filtrate and record this weight on the extraction log.

2) Assemble the filtratation device, place and secure the filter on the screen.

3) Weigh a 100 g subsample of the waste and record the weight on the extraction log. Allow slurry samples to stand for a sufficient time to allow the solid phase to settle.

   Note: If necessary, centrifuge the waste prior to filtration. If centrifugation is performed, decant and filter the liquid and then filter the solid portion of the waste through the same filtration setup.

4) Quantitatively transfer the entire waste sample to the filter holder. Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, allow the sample to warm to room temperature in the device before filtering.

   If any waste material (>1% of original sample weight) has adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the weight of the subsample in order to determine the actual weight of waste sample that will be filtered.

5) Gradually apply vacuum or gentle pressure of 1-10 psi, until the pressurizing gas (nitrogen) moves through the filter. If this point is not reached under 10psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10 psi increments in 2-minute intervals to a maximum of 50 psi. Proceed to the next 10 psi increment only if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval. Stop the filtration when the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 40 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period).

   NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

6) The material that remains in the filter holder is defined as the solid phase of the

waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will contain some material that appears to be a liquid. This material may not filter even after applying vacuum or pressure filtration. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

Determine the weight of the liquid phase (LP) by subtracting the weight of the filtrate container (FC) from the total weight of the filtrate-filled container (FF). Determine the weight of the solid phase (SP) by subtracting the weight of the liquid phase (LP) from the weight of the total waste (SW) sample.

Calculate percent solids:

Determine the weight of the Liquid Phase (LP): (FF)-(FC)
Determine the weight of the Solid Phase (SP): SW-LP

Percent solids = $(SP \div SW) * 100$

Where:
SP= Weight of solid
SW= Total weight of waste

7) If the calculated percent solids are less than 0.5%, proceed to Section 11.3.

If the calculated percent solids are equal to or greater than 0.5%, proceed to Section 11.1.3 for preliminary determination for particle size reduction unless you observe that a small amount of the filtrate is entrained in wetting of the filter. In which case, determine the percent dry solids:

Remove the solid phase and filter from the filtration apparatus. Dry the filter and solid phase in a drying oven maintained at a temperature of 100 °C (± 20°C) until two successive weighing yield the same value within ±1%.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

Record the final weight and calculate percent dry solids:

Percent dry solids $=[((W_d) - F_t) \div SW] * 100$

Where:
W_d = Weight of dry waste and filter

F_t = Tared weight of filter
SW= Initial weight of waste

If the percent dry solids are less than 0.5%, proceed to Section 11.3.

If the percent dry solids are greater than or equal to 0.5%, obtain a fresh portion of waste and proceed to section 11.1.2 for preliminary determination for particle size reduction.

Particle Size Reduction

Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid can pass through a standard 9.5 mm sieve.

If the surface area is smaller or the particle size larger than described above, crush, cut or grind the waste to a surface area or particle size as described above.

Note: Wastes and appropriate reduction equipment should be refrigerated, if possible, at 4°C prior to particle size reduction and the means used to affect the particle size reduction must not generate heat. If reduction of the waste is required, exposure of the waste to the atmosphere should be minimized to the extent possible.

Proceed to Section 11.1.3 for determination of the extraction fluid.

Extraction Fluid Determination

Weigh out a small subsample of the solid phase of the waste, if necessary reduce the solid to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 g of the solid phase of the waste to a 400 mL beaker or Erlenmeyer flask.

Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH.

If the pH is <5.0, use extraction fluid #1 and proceed to Section 11.2.

If the pH is >5.0, add 3.5 mL 1N HCl, stir briefly, cover with a watchglass, heat to 50°C, and hold at 50°C for 10 minutes. Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 11.2.

TCLP Extraction Procedure

Filtration

If the waste will obviously yield no liquid when subjected to pressure filtration (100% solid) weigh out a 100 g subsample of the waste and proceed to Section 11.2.2.

If the waste is liquid or multiphasic, separate the liquid phase from the solid using the filtration procedure outlined in Section 11.1.1, Steps 1-6.

If the percent solids of the filtered waste as determined from Section 11.1.1 is <0.5% dry solids proceed to Section 11.3 for preparation of the TCLP extract.

If the percent solids of the filtered waste as determined from Section 11.1.1 is $\geq$ 0.5% dry solids and if the result of the preliminary evaluations indicated particle size reduction is needed (11.1.2), perform particle size reduction, then quantitatively transfer the solid material into the extractor bottle along with the filter that was used to separate the initial liquid from the solid phase and proceed to Section 11.2.2.

If the filtered waste does not require particle size reduction, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 11.2.2.

Extraction

Determine the amount of extraction fluid to add to the extractor vessel:

Weight of Extraction Fluid = $(20 * S_{\%} * Sp) \div 100$

Where:
$S_{\%}$ = Percent solids
Sp= Weight of waste filtered

Slowly add the amount of appropriate extraction fluid to the extractor vessel. Close the extractor bottle tightly, secure in the rotary agitation device, and rotate at 30rpm (±2) for 18 ± 2 hours. The ambient temperature of the room during the extraction period should be 23°C (±2°C). Record the room temperature and the rotation check on the benchsheet.

After extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

TCLP Extract Preparation

If the waste was less than 0.5 percent solid, the waste after filtration (11.1.1. or 11.2.1) is defined as the TCLP extract.

If the waste contained no initial liquid phase (percent solid 100%) the filtered liquid material obtained after extraction (11.2.2) is defined as the TCLP extract.

If the waste was greater than or equal to 0.5 percent solids, and if compatible, combine the filtered liquid resulting from extraction (11.2.2) with the initial liquid phase of the waste obtained from 11.2.1. The combined liquid is defined as the TCLP extract.

If the filtered initial liquid phase of the waste (11.2.1) is not compatible with the filtered liquid material obtained after extraction (11.2.2), do not combine the liquids. Prepare and analyze the liquids, collectively defined as the TCLP extract, separately and combine the results from analysis mathematically (See Section 11.5.)

Following collection of the TCLP extract, measure and record the pH of the extract. Immediately aliquot and preserve the extract for analysis. Acidify metals aliquots with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, do not acidify the remaining portion of the extract and analyze the extract as soon as possible. Store all TCLP extracts under refrigeration (4°C) until extraction and/or analysis.

Non-Volatile Preparative Extraction of TCLP Extracts

TCLP Extracts are solvent extracted by separatory funnel following the approved laboratory SOP for Method 3510C. The extraction fluid (fluid blank) that was extracted with the samples is used for the method blank. The laboratory control sample (LCS) is extracted using the tumbled Extraction Fluid.

The following volumes are extracted:

Analyte Category	Method	Extraction Volume (mL)	Final Volume (mL)
Pesticides	8081	100	10.0
Herbicides	8151	20	10.0
BNAs	8270	200	1.0

Metals Digestion of TCLP Extracts

TCLP extracts to be analyzed for metals are acid digested according to laboratory SOP LM-MP-3010 except in those instances where digestion causes loss of metallic analytes and analyzed following laboratory SOP LM-MI-6010.

Analysis

The TCLP extracts (after extraction, digestion) are analyzed following the laboratory SOP for the determinative method requested. If individual phases are analyzed separately the results are combined mathematically using a volume weighted-average:

Final Analyte Concentration = $[(V_1)(C_1) + (V_2)(C_2)] \div (V_1 + V_2)$

Where:
V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

12.0. CALCULATIONS

Not applicable

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1. The TCLP extraction bench sheet is completed by the analyst(s) that performed the procedure and reviewed by the Department Supervisor. Problems encountered during the extraction process are documented using a nonconformance report that records the root-cause, action taken, and the result of action taken. Data that does not meet the minimum acceptance criteria is flagged using data qualifiers and a description of the outage is written in the case narrative provided with the data package report.

14.0 METHOD PERFORMANCE

14.1. An Initial Demonstration of Capability is required for each analyst before unsupervised performance of this method.

14.2. A Method Detection Limit (MDL) determination for each test method referenced in this SOP is performed following the procedure described in the reference method, 40CFR, Part 136, Appendix B and laboratory SOP LP-LB-009. The MDL is verified or repeated when a significant change to the method occurs. Significant changes include the use of alternate reagents or standard reference materials, new instrumentation or the use of alternate sample preparation procedures.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Waste Solvents
- Solid Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

16.1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final

Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

17.0 TABLES, DIAGRAMS & FLOWCHARTS

17.1 Table 1: Primary Materials Used

17.2 Appendix A: Terms and Definitions

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

STANDARD OPERATING PROCEDURE
VOLATILE ORGANIC COMPOUNDS BY GC/MS
SW-846 8260B
Applicable Matrices: Non-Potable Water and Solid and Chemical Materials
Standard Compound List and Reporting Limits: See Table 1

APPROVAL SIGNATURES

Laboratory Director: Christopher A. Ouellette — Date: December 12, 2005

QA Manager: Kirstin L. McCracken — Date: December 12, 2005

Organics Manager: Jennifer L. Clements — Date: December 12, 2005

Proprietary Information Statement:

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the GC/MS procedure for the analysis of volatile organic compounds in ground and surface water, waste solvents, oily wastes, soils and sediments using a chromatographic column with a temperature program to separate the desorbed purgeables followed by mass spectral detection. This SOP is applicable to the analytical procedure only. The techniques by which compounds may be introduced into the GC/MS system are described in the following SOPs:

LM-MV-5030	Purge and Trap of Aqueous Samples
LM-MV-5035	Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples.

1.2 The analytes that can be determined by this procedure and their associated Reporting Limits (RL) are listed in Table 1, Section 18.

2.0 SUMMARY OF METHOD

2.1 Basic Principles - The analytes are introduced into the GC/MS by purge-and-trap techniques (Method 5030 or Method 5035). Upon desorption from the trap, the volatile compounds are introduced directly to a wide-bore capillary column. A temperature program is used to separate the purgeables. The eluted analytes pass through a jet separator and are carried on the gas stream into the ion source of a mass spectrometer. The ionized molecules are focused and separated according to their mass/charge (m/z) ratio by the quadrupole analyzer. The signal is amplified by an electron multiplier and interpreted by the mass spectrometer data system to produce a total ion chromatogram and mass spectra for every data point on the chromatogram.

2.2 General Method - The mass spectrometer is calibrated to recognize m/z values in the range of 35-300 amu. Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. Analytes are quantified using internal standard calibration. The concentration of each identified component is measured by relating the MS response of the quantification ion produced by that compound to the MS response of the quantification ion produced by a compound that is used as an internal standard. The performance of the mass spectrometer is verified by the injection of 4-Bromofluorobenzene (BFB). Next, the instrument must demonstrate acceptable chemical calibration and linearity by the analysis of five concentrations of a standard mix containing the analytes of interest, as well as the surrogates and internal standards. Before any samples are analyzed, a method blank must be analyzed to demonstrate that the instrument is free from contamination, and that surrogate recovery criteria are met. All analyses must occur within 12 hours of the injection of the passing BFB. Another analytical sequence may be started by the analysis of a passing BFB MS tune followed by a continuing calibration standard.

2.3 This procedure is based on Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (Method 8260B), Revision 2, December 1996, USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

3.0 DEFINITIONS

A list of terms and definitions is given in Appendix C.

4.0 INTERFERENCES

4.1 During analysis, major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of Teflon tubing, Teflon thread sealants, or flow controllers with rubber components in the purging device should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of laboratory reagent blanks provide information about the presence of contaminants. Subtracting blank values from sample results is not permitted.

4.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. The auto-sampler utilizes a single purge vessel that is automatically rinsed between analyses. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagent blanks may be analyzed to check for carry-over.

4.3 Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride; otherwise, random background levels will result. Since methylene chloride will permeate Teflon tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory worker's clothing should be cleaned frequently since clothing previously exposed to methylene chloride fumes during common extraction procedures can contribute to sample contamination. Extraction laboratory personnel should not enter the volatile analytical laboratory.

4.4 Traces of ketones, methylene chloride, and some other organic solvents can be present even in the highest purity methanol. This is another potential source of contamination, and should be assessed before standards are prepared in the methanol.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichlorethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride.

5.3 Primary Materials Used

Table 3, Section 18 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **Note: The table does not include all materials used in the procedure. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used can be found in Section 7. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

- Methanol

6.0 EQUIPMENT AND SUPPLIES

6.1 Containers

- Sample Storage Containers: 40 mL screw cap vials equipped with Teflon faced silicone septum, certified clean, known volume of 44 mL (also see Method 5035).
- Standard Storage Containers: 1-5 mL Mininert vials with Teflon lined screw caps

6.2 Computer Hardware/Software: GCMS Acquisition Platform - Hewlett-Packard ChemStations. Data Processing - Hewlett-Packard 9000-series computers, an HP9000 D250 (Chemsvr4) and an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5.

6.3 Instrumentation

- VOA Autosampler: Tekmar ALS 2050, EST Archon, or equivalent
- Purge & Trap: Tekmar LSC 2000, EST Encon; VOCARB 3000 trap or equivalent
- Gas Chromatograph: Hewlett-Packard 5890 Series II and 6890
- Mass Spectrometer: Hewlett-Packard 5971 MSD, Hewlett-Packard 5973 MSD
- Primary Column: Fused silica capillary column, J&W DB624 75 m x 0.53 mm x 3.0 um or equivalent

6.4 Syringes: 250 µL - 10 mL gas tight hypodermic syringes with Luer-Lok tip, Micro syringe 10 - 100 µL

7.0 REAGENTS AND STANDARDS

7.1 Trap Packing Materials - VOCARB 3000 or equivalent traps may be used, following the manufacturer's instructions.

7.2 Reagents

- Methanol - Purge and Trap Grade, demonstrated to be free of analytes.
- Reagent water - Deionized water is filtered using a Milli Q plus ™ filtration system and then boiled for one hour, and purged with helium for a minimum of fifteen minutes. The water is stored in clean, narrow-mouth bottles with Teflon lined septa and screw caps.
- Hydrochloric acid (1:1) - Measured volumes of conc. HCl are carefully added to an equal volume of reagent water.
- Sodium Bisulfate ($NaHSO_4$) Solution - 20% wt/v. Preservative for soil samples (5035).

7.3 Standards

7.3.1. Stock Standard Solutions - These solutions are purchased as certified solutions or prepared from pure standard materials. Commercial standards arrive ampulized in concentrations ranging from 1-5 mg/mL. Preparation of Working Standards from the stock standards is outlined in Appendix A.

7.3.2. Preparation of Calibration Standards

Prepare the five-point calibration curve for the waters and soils using the Working Standards prepared in Appendix A. The volatile water curve is prepared in 44 mL vials; all standards are spiked directly through the septum of the 44 mL vial. Prepare the routine level water and medium level soil curve as follows:

ROUTINE LEVEL WATER AND MEDIUM LEVEL SOIL CALIBRATION CURVE					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
8260 Calibration Standard - Mixed	2.2 µL	8.8µL	22 µL	44 µL	88 µL
8260 Calibration Gas - 100 mg/L	2.2 µL	8.8µL	22 µL	44 µL	88 µL
8260 Calibration Added - 100 mg/L	2.2 µL	8.8µL	22 µL	44 µL	88 µL
Internal Standard - 50 mg/L	44 µL	44 µL	44 µL	44 µL	44 µL
Final Volume	44 mL	44 mL	44 mL	44 mL	44 mL
FINAL CONCENTRATION in µg/L					
All analytes except as listed below:	5	20	50	100	200
1,4-Dioxane	250	1000	2500	5000	10000
Isobutyl alcohol	250	1000	2500	5000	10000
Propionitrile	20	80	200	400	800
Tetrahydrofuran	50	200	500	1000	2000
Internal Standards	50	50	50	50	50

As chloroethane often follows a quadratic, rather than linear, pattern, a sixth point may be prepared and analyzed for the calibration gases for this purpose. Even though all the gases will be contained in that calibration point, it is only anticipated that the chloroethane value will be used.

Prepare the low-level soil curve in 44 mL vials containing 5 mL of VOA free water as follows:

LOW LEVEL SOIL CURVE					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
8260 Calibration Standard - Mixed	2.2 µL	1.0µL	2.5 µL	5.0 µL	10 µL
8260 Calibration Gas - 100 mg/L	2.2 µL	1.0µL	2.5 µL	5.0 µL	10 µL
8260 Calibration Added - 100 mg/L	2.2 µL	1.0µL	2.5 µL	5.0 µL	10 µL
Internal Standard - 50 mg/L	44 µL	5 µL	5 µL	5 µL	5 µL
Final Volume	44 mL then transfer 5 mL to 44 mL vial	5 mL	5 mL	5 mL	5 mL
FINAL CONCENTRATION in µg/L					
All analytes except as listed below:	5	20	50	100	200
1,4-Dioxane	250	1000	2500	5000	10000
Isobutyl alcohol	250	1000	2500	5000	10000
Propionitrile	20	80	200	400	800
Tetrahydrofuran	50	200	500	1000	2000
Internal Standards	50	50	50	50	50

Prepare the five-point calibration curve for Low Level Waters as follows:

LOW LEVEL WATER CALIBRATION CURVE					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
8260 Calibration Standard Low - Mixed	1.8 µL	8.8µL	17.6 µL	44 µL	88 µL
8260 Calibration Gas Low - 25 mg/L	1.8 µL	8.8µL	17.6 µL	44 µL	88 µL
8260 Calibration Added Low - Mixed	1.8 µL	8.8µL	17.6 µL	44 µL	88 µL
Internal Standard Low - 25 mg/L	8.8 µL	8.8 µL	8.8 µL	8.8 µL	8.8 µL
Final Volume	44 mL	44 mL	44 mL	44 mL	44 mL
FINAL CONCENTRATION in µg/L					
All analytes except as listed below:	1	5	10	25	50
1,4-Dioxane	51	250	500	1250	2500
Isobutyl alcohol	51	250	500	1250	2500
Propionitrile	4	20	40	100	200
Tetrahydrofuran	14	70	140	350	700
Acrolein	5	25	50	125	250
Acetone	5	25	50	125	250
4-Methyl-2-pentanone	5	25	50	125	250
Methyl ethyl ketone	5	25	50	125	250
Tetrahydrofuran	5	25	50	125	250
2-Hexanone	5	25	50	125	250
Internal Standards	5	5	5	5	5

Additionally, for manual injections these standards may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above.

7.3.3. Preparation of Initial Calibration Verification (ICV) – Second Source Standard

The ICV must be obtained from a different source than that which is used to prepare the calibration curve, or if one is not available, a second lot of the same manufacturer. The volatile water ICV is prepared in 44 mL vials. All standards are spiked directly through the septum of the 44 mL vial. Prepare the routine level water and medium level soil ICV as follows:

ROUTINE LEVEL ICV	
8260 Calibration Standard - Mixed	22 µL
8260 Calibration Gas - 100 mg/L	22 µL
8260 Calibration Added - 100 mg/L	22 µL
Internal Standard - 50 mg/L	44 µL
Surrogate Standard - 50 mg/L	44 µL
Final Volume	44 mL
FINAL CONCENTRATION in µg/L	
All analytes except as listed below:	50
1,4-Dioxane	2500
Isobutyl alcohol	2500
Propionitrile	200
Tetrahydrofuran	500
Internal Standards	50

Prepare the low-level soil ICV as follows:

LOW LEVEL SOIL ICV	
8260 Calibration Standard - Mixed	2.5 µL
8260 Calibration Gas - 100 mg/L	2.5 µL
8260 Calibration Added - 100 mg/L	2.5 µL
Internal Standard - 50 mg/L	5 µL
Surrogate Standard - 50 mg/L	5 µL
Final Volume	5 mL
FINAL CONCENTRATION in µg/L	
All analytes except as listed below:	50
1,4-Dioxane	2500
Isobutyl alcohol	2500
Propionitrile	200
Tetrahydrofuran	500
Internal Standards	50

Prepare the low level water ICV as follows:

LOW LEVEL WATER ICV	
8260 Calibration Standard Low - Mixed	17.6 µL
8260 Calibration Gas Low - 25 mg/L	17.6 µL
8260 Calibration Added Low - Mixed	17.6 µL
Internal Standard Low - 25 mg/L	8.8 µL
Surrogate Standard - 25 mg/L	8.8 µL
Final Volume	44 mL
FINAL CONCENTRATION in µg/L	
All analytes except as listed below:	10
1,4-Dioxane	500
Isobutyl alcohol	500
Propionitrile	40
Tetrahydrofuran	140
Acrolein	50
Acetone	50
4-Methyl-2-pentanone	50
Methyl ethyl ketone	50
Tetrahydrofuran	50
2-Hexanone	50
Internal Standards	5

Additionally, for manual injections these standards may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above.

7.3.4. Preparation of Continuing Calibration Verification (CCV)

CCVs are prepared in the same manner as the calibration standards using the same source that is used to prepare the calibration curve. Prepare the CCVs for routine level water and medium level soil, low-level soil, and low-level water exactly as the Level 2, 3 or 4 standard is prepared in Section 7.3.2.

Additionally, for manual injections these standards may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above and injected into the purge vessel.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Sample collection and preservation for aqueous samples is described in STL SOP LM-MV-5030 and for solid samples in STL SOP LM-MV-5035.

8.2 Unless specified by contract, other regulation, or Quality Assurance Project Plan, the volatile holding times as specified by method and federal regulation are as follows:

Sample Type	Holding Time
Aqueous Preserved (HCl to pH<2)	14 days from collection
Aqueous Non-Preserved	7 days from collection
TCLP Leachate	14 days from leaching
Soil in Sodium Bisulfate solution	14 days from collection
Soil in Encore Device	Extruded into sodium bisulfate within 48 hours, analyzed 14 days from collection
Methanol Extract of Soil	14 days from collection
Sludge	14 days from collection

8.3 Samples are stored at $4^oC \pm 2^oC$ in a storage area free of organic solvent vapors and direct or intense light. The pH of samples is recorded, and samples may be screened.

8.4 Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 The minimum frequency requirements, acceptance criteria and recommended corrective action for all QC samples are summarized in Section 18, Table 5. Below is a summary of each type of QC sample that is analyzed with the method.

9.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each batch of 20 or fewer samples run within a 12-hour window. These samples show that the laboratory is in control, independent of the sample matrix.

9.3 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each batch of 20 samples. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.4 Surrogate standards are added to all field and QC samples before preparation and/or analysis to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.5 Internal Standards – All samples are spiked with internal standards as described in Section 7.

9.6 Instrumental QC standards include a Bromofluorobenzene (BFB) every 12 hours, before each Initial Calibration (ICAL) and CCV. A five-point ICAL is generated for each target analyte. After the ICAL, an ICV standard, also referred to as a second source standard, is analyzed to verify the ICAL standard formulation. CCV standards are analyzed every 12 hours immediately following the BFB.

10.0 CALIBRATION AND STANDARDIZATION

10.1 4-Bromofluorbenzene (BFB) - Prior to the acquisition of a calibration curve or the analysis of samples, a 2 μL aliquot of BFB (25 μg/mL) is manually introduced into the GC. The data processing system acquires and averages three scans (apex scan, scan prior, and scan preceding) and performs background subtraction of the single scan prior to the elution of the BFB. The BFB must meet the criteria in Table 2 before initial or continuing calibration may proceed.

10.2 Samples may be run for 12 hours after successful initial and/or continuing calibration. The official start time of the 12-hour window is the time of the BFB injection. The last sample in the window must be injected within 12 hours of that time.

10.3 Initial Calibration – A five-point calibration curve is analyzed at the concentrations and in the manner specified in Section 7.3.2.

10.4 For each target analyte and surrogate, calculate the mean Response Factor (RF) from analyses of the five calibration solutions. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (%RSD - see Appendix B for calculations).

The following criteria must be met for a calibration to be considered acceptable:

- System Performance Check Compounds (SPCCs) must meet the following minimum mean RF: Chloromethane, 1,1-Dichloroethane, and Bromoform - 0.10; Chlorobenzene and 1,1,2,2-Tetrachloroethane – 0.30.
- The following Calibration Check Compounds (CCCs) must have a %RSD of ≤ 30%: 1,1-Dichloroethene, Toluene, Chloroform, Ethylbenzene, 1,2-Dichloropropane, and Vinyl chloride. If the CCCs are not included in the list of analytes, then the criteria in Section 10.5 should be used.
- The Relative Retention Time (RRT) for each target analyte in each calibration standard should agree within 0.06 RRT units (See Appendix B for calculation).

10.5 If the %RSD ≤ 15%, for all analytes, the calibration is acceptable and the mean RF may be used for quantification. If this criterion is not met, either use another suitable quantification method, or correct the problem and repeat the calibration.

10.6 Alternate Quantification. In some cases, it may be preferable to use either linear regression or Quadratic Equations to quantify the compounds. The following approaches may be used:

Linear Regression - A curve of concentration vs. peak area is generated for each analyte and the correlation coefficient is calculated. The calibration must have a correlation coefficient (r) ≥ 0.99 (0.995 for DoD) for acquisition of samples to continue. The use of linear regression requires a minimum of 5 calibration points. See SW-846 Method 8000B for linear regression calculations.

Quadratic Equation - For some compounds, the response is not linear, and in this case a quadratic equation may be employed. For those compounds, the coefficient of determination (r^2) is generated. The coefficient of determination must be $\geq$ 0.99 for acquisition to continue. The uses of quadratic equations require a minimum of 6 calibration points for second order regression and 7 points for third order regression. As chloroethane often follows a quadratic, rather than a linear, pattern, It is anticipated that a sixth point will be analyzed containing only the gases, and that only the chloroethane point is going to be used from this standard. See SW-846 Method 8000B for quadratic equation calculations.

Once a method of calibration is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is generated.

10.7 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV. The calculated concentration of each analyte must be within $\pm$ 25% of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate.

If after successful analysis of the ICV time remains in the 12 -hour analytical window samples may be analyzed without analysis of a CCV; otherwise a CCV must be performed.

10.8 Continuing Calibration Verification (CCV)

At the beginning of each 12-hour shift in which samples are to be run, analyze a BFB as outlined in Section 10.1. A CCV standard, at or below mid-calibration range, is analyzed at the beginning of each 12-hr work shift. The concentration of the CCV is varied.

Calculate the RF and percent difference or drift (see Appendix B for calculation) for each target analyte and surrogate standard. The SPCCs must meet the criteria outlined in Section 10.3. The percent difference or drift for the CCCs must be $\leq$ 20%. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

In addition, the internal standard retention time should not change by more than 30 seconds from the retention time in the mid-point standard of the most recent ICAL. The extracted ion current profile (EICP) area of the internal standards in the calibration verification standard should not change by more than a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent ICAL.

If the CCV fails, it may be repeated once. If it still fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples analyzed after a failing CCV must be reanalyzed, unless the analyte in the CCV is high and that analyte is not detected in the associated samples.

10.9 Troubleshooting: the following items can be checked in case of calibration, QC or instrument failures:

- Chloromethane response can be low if the purge flow is too fast.
- Bromoform response can be low if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantification ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.
- Contaminated transfer lines in purge-and-trap systems and/or active sites in the trap can degrade the response of Tetrachloroethane and 1,1-dichloroethane.
- 2-Chloroethylvinylether response can be drastically affected/suppressed by soil, foam, or other artifacts contaminating the inside of the soil purge needle. It is also susceptible to active sites/contamination anywhere in the helium path from the autosampler (soils) to the injection port on the GC.
- If the response of the later eluting compounds is low, especially with soils, the purge flow may have been reduced by an obstruction in the helium flow path.
- Poor chromatography and response of the gases are often the result of incorrect placement of the column head in the injection port and/or contamination of the first 6-10 inches of the column from samples and small pieces of injection port septum.
- Erratic response of various compounds and unstable calibrations can be the result of a worn out/contaminated purge trap. Variable matrices such as tissues, soils and moderately foamy samples can be the cause. Samples high in late eluting hydrocarbons or sulfur dioxide will also degrade the trap.

11.0 PROCEDURE

11.1 The following samples are routinely prepared with each batch of samples to be run within a 12 hour window:

11.1.1. Laboratory Method Blanks

For routine level waters, a 44 mL sample vial is filled with reagent water (no air bubbles). Internal and surrogate standards are added separately by the injection through the septum of 44 µL of each of the 50-mg/L internal and surrogate standards.

For medium level soils, a 44 mL sample vial is partially filled with reagent water, 880 µL of methanol is added to it, and the vial is filled with reagent water (no air bubbles). Internal and surrogate standards are added separately by the injection through the septum of 44 µL of each of the 50-mg/L internal and surrogate standards.

For the low-level waters, a 44 mL sample vial is filled with reagent water (no air bubbles). Internal and surrogate standards are added separately by the injection through the septum of 8.8 µL of each of the 25-mg/L internal and surrogate standards.

For low-level soils, a 44 mL sample vial containing 5 gms of sand, 5 mLs of reagent water, and a stir bar is spiked with 5 µL of each of the 50-mg/L internal and surrogate standards.

11.1.2. Laboratory Control Sample (LCS)

For routine level waters and medium level soils, a 44 mL sample vial is filled with reagent water (no air bubbles). Internal and surrogate standards are added separately by the injection through the septum of 44 µL of each of the 50-mg/L internal and surrogate standards, and target analytes are added by injection through the septum of 22 µL of each of the Calibration Standard Mix, Calibration Gas, and Calibration Added standards.

For the low-level waters, internal and surrogate standards are added separately by the injection through the septum of 8.8 µL of each of the 25-mg/L internal and surrogate standards, and target analytes are added by injection through the septum of 17.6 µL of each of the Calibration Standard Mix Low, Calibration Gas Low, and Calibration Added Low standards.

For low-level soils, 5 grams of clean sand is added to a 44 mL sample vial containing a stir bar. The vial is spiked through the septum with 5 µL of each of the 50-mg/L internal and surrogate standards, and 2.5 µL of each of the Calibration Standard Mix, Calibration Gas, and Calibration Added standards.

Additionally, for manual injections the LCS may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above.

11.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

MS/MSD are analyzed with each batch of 20 client specific samples of the same matrix. If there is no designated MS/MSD in a batch, one will be prepared if the extra volume for doing so is provided by the client. If not, this is noted as a non-conformance. For DoD, project specific MS/MSD must be run in each batch.

For routine and low-level water samples, and medium level soils, the MS/MSD are prepared exactly as the LCS is prepared in Section 7.5, except that instead of spiking 44 mL of reagent water, a client sample is spiked.

For low-level soil samples, the MS/MSD are prepared by spiking a vial with 5 gms of client sample and 5 mLs of sodium bisulfate solution with 5 µL of each of the 50-mg/L internal and surrogate standards, and 2.5 µL of each of the Calibration Standard Mix, Calibration Gas, and Calibration Added standards through the septum.

Additionally, for manual injections these standards may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above.

11.2 Each sample to be analyzed is spiked with internal and surrogate standards. For routine level water and medium level soil samples. Internal and surrogate standards are added separately by the injection through the septum of 44 μL of each of the 50-mg/L internal and surrogate standards. For low level soils, internal and surrogate standards are added separately by the injection through the septum 5 μL of each of the 50-mg/L internal and surrogate standards For low-level waters, internal and surrogate standards are added separately by the injection through the septum of 8.8 μL of each of the 25-mg/L internal and surrogate standards.

11.3 Cleaning blanks (CBLK) consisting of VOA free water may be analyzed after high-level samples at the discretion of the analyst.

11.4 Sample Introduction and Purging - See preparation and introduction Methods 5030 and 5035.

11.5 Gas Chromatography/Mass Spectrometry - Data is acquired and stored over the nominal mass range of 35-300 atomic mass units (amu) with a total cycle time (including scan overhead time) of one second at 70 electron volts. The cycle time is adjusted to measure five or more spectra during the elution of each GC peak. A multi-stage temperature ramp is used to separate the components of interest for this analysis. A typical GC temperature program is described below, but is subject to change at the discretion of the analyst:

Initial temperature:	40° C
Initial time:	4 min.
Ramp1:	7° C/min. to 100° C.
Ramp2:	4.2° C/min. to 120° C, hold for 0 min.
Ramp3:	28° C/min. to 220° C, hold for 2.1 min.
Carrier Gas:	Helium

11.6 Instrument control and acquisition parameters are defined on the ChemStation software for each instrument. Arrange the samples in the autosampler. Acquire the data and evaluate the results to confirm qualitative identification and quantification.

11.7 The data system tentatively identifies target analytes by comparing the retention time of the peaks to a window set around the daily calibration standard, and searches in that area for the primary and up to two secondary ions characteristic of the target analyte. All tentative identifications made by the computer are reviewed and either accepted or rejected by the analyst and/or data reviewer using the following criteria:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the user-created database. In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance if a tentative identification is to be made, and should be evaluated even if they are below 10% relative abundance.

- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

11.8 Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. Because purgeable organic compounds are relatively small molecules and produce comparatively simple mass spectra, this is not a significant problem for most method analytes.

11.9 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs. Two of the three isomeric xylenes are examples of structural isomers that are not resolved on the capillary column. These groups of isomers will be reported as isomeric pairs.

11.10 Tentatively Identified Compounds (TICs)

TICs may be reported upon client request. In general, TICs whose peak heights are > 10% of the nearest internal standard (>40% for low level water analysis) may be reported.

Perform the library search, and visually compare the sample spectra with the nearest library search and assign a tentative identification. The library search should not include peaks that are < 10% of the nearest internal standard, target analytes, or peaks that elute earlier than 30 seconds before the first target analyte.

The following criteria are used in qualitatively identifying these compounds:

- Relative intensities of ions greater than 10% of the most abundant ion in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.

- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

The laboratory may determine that a more general identification can be made, such as “unknown alkane, naphthalene derivative, unknown aldehyde, etc.”. TIC concentrations are calculated as outlined in Appendix B using an RF of 1.00. All TICs concentrations are reported with a “J” qualifier to indicate that the quantification is estimated. All cases of tentative identification are flagged with an “N” to indicate that there is presumptive evidence of a compound.

11.11 Quantification of Target Analytes

After a compound has been identified, the data system quantifies the concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP using the equations given in Appendix B. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are required, include this information in the non-conformance report and project narrative.

11.12 If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP LP-LB-0006 *Manual Integration.*

11.13 After analysis is complete, evaluate the results against the performance criteria given in Section 10 and Table 3, Section 18 and perform corrective action as necessary.

11.14 Review the samples for carry-over from high-level samples run just prior to the sample for any sign of carry over. Re-analyze the sample is carry-over is suspected.

11.15 Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should ideally result in a determination within the upper half of the calibration curve.

12.0 CALCULATIONS

See Appendix B.

13.0 DATA ASSESSMENT, CORRECTIVE ACTION & REPORTING

13.1 Review the samples, standards and QC samples against the acceptance criteria in Table 4. If the results do not fall within the established limits, perform the recommended corrective action. If corrective action is unsuccessful, document the situation with a nonconformance report and/or qualify the data using an appropriate data qualifier (see

Appendix C for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

13.2 In the absence of project specific requirements, use the control limits specified in Table 4. The control limits in Table 1 are based on in-house statistically generated limits. In some cases, the in-house limits were outside of Department of Defense (DoD) limits as specified in the Quality Systems Manual for Environmental Laboratories. Where this is the case, the laboratory uses the stricter, DoD limits that are presented in bold in Table 4. For DoD projects, the in-house laboratory limits are also included in the project report.

Based on the number of analytes in the LCS (70-91), it is statistically likely that at least four analytes will marginally exceed the control limits; therefore, 4 marginal exceedances are allowed. A marginal exceedance (ME) is defined as being outside of the control limit of $\pm$ 3 SD, but within $\pm$ 4 SD. In order to easily calculate these limits, the following equation may be used:

$$\text{ME (Lower Limit)} = \text{Lower Limit} - \frac{(\text{Upper Limit} - \text{Lower Limit})}{6}$$

$$\text{ME (Upper Limit)} = \text{Upper Limit} + \frac{(\text{Upper Limit} - \text{Lower Limit})}{6}$$

In addition, the following analytes have been identified as poorly performing analytes based on statistical data accumulated by the laboratory: acrolein in the medium level water and medium level soil matrices, and methyl iodide in the low level water matrix. Decisions regarding batch acceptability are not based on those analytes in the specific matrix identified.

13.3 Data Reporting

The laboratory's RL for each target analyte is provided in Table 1. Report the data to the RL adjusted for sample matrix, percent moisture, and sample dilution/concentration. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results that have concentrations for a target analytes less than the RL with a "U" qualifier. Unless otherwise specified, report the results for solid matrices on a dry weight basis.

Some projects may require reporting positively identified target analytes less than the RL. In this case, the analyte can be qualitatively detected but not accurately quantified. Flag all results less than the RL with a "J" data qualifier.

Some projects may require RLs that are less than the laboratory's routine RL. Sample results may be reported to the project RL if the project RL is greater than the Quantification Limit (QL) and above the MDL. In this context, the QL is defined as the concentration of the low calibration standard. If the project RL is less than the QL, all values less than the QL must be reported as estimated and qualified with a "J".

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-LB-009 *Determination of Method Detection Limits.*

13.4 Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample
D = Compound is identified in an analysis at a secondary dilution factor
E = Compound quantification is above the instrument's calibration range for this analysis
J = Indicates an estimated quantification value
U = Compound was analyzed for but not detected
X = The reported compound is a suspected laboratory contaminant
Y = an additional qualifier which will be defined at the time of use by the data reviewer
Z = The reported result is based on the combined responses from coeluting compounds
* = Data outside of control limits

13.5 Data Management and Records: All electronic and hardcopy data is managed, retained, and archived as specified in laboratory SOP LP-QA-0014 *Laboratory Records*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.4 The laboratory statistically derived control limits used to evaluate accuracy, precision and surrogate recoveries are provided in Table 2. The control limits for accuracy are based on compiled data and are set at 3 standard deviations around the mean using the procedures described in laboratory SOP LP-QA-012 *Control Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 The following waste streams are produced when this method is carried out.

- Aqueous Waste
- Solvent Waste
- Solid Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (Method 8260B), Revision 2, December 1996, USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

17.0 SOP REVISION HISTORY

The following changes were made in this revision:

Section 6:	Added computer hardware and software.
Section 7:	7.1 - Removed solvents not used in analytical method.
Section 10:	10.3 - Added additional quantification options. 10.4 - 10.6 Added detail about repeating CCV. 10.7 Added Troubleshooting.
Section 12:	Moved calculations to Appendix B. Moved data reporting to Section 13.
Section 13:	13.1 - Added detail regarding the use of DoD LCS and Surrogate Limits. 13.3 - Added SOP reference for Data Management & Records.
Section 14:	Completely revised section.
Section 17:	New Section added.
Table 1:	Added Footnotes.
Table 3:	Changed Table 3 from Calibration Criteria to Primary Materials Used.
Table 4:	Combined Table 4, 5, and 6, updated control limits, added Low Level Water and Soil Control Limits, added footnotes.
Appendix B:	New Appendix added containing all calculations.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

Table 1:	Target Analyte List, Chemical Abstract Services Numbers and Reporting Limits
Table 2:	BFB Key Ions and Ion Abundance Criteria
Table 3:	Primary Materials Used
Table 4:	Control Limits as Accuracy (%R) and Precision (RPD)
Table 5:	QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action
Appendix A:	Standard Preparation Tables
Appendix B:	Equations
Appendix C:	Terms & Definitions

Table 1: Target Analyte List, Chemical Abstract Services Numbers and Reporting Limits

Analyte	CAS No.	Reporting Limit[1]		
		Water or Soil µg/L or µg/Kg	Low Water µg/L	Methanol Extracts[2] µg/Kg
Acetone	67-64-1	5.0	5.0	500
Acrolein	107-02-8	5.0	5.0	500
Acrylonitrile	107-13-1	5.0	1.0	500
Allyl Chloride	107-05-1	5.0	1.0	500
Benzene	71-43-2	5.0	1.0	500
Bromobenzene	108-86-1	5.0	1.0	500
Bromochloromethane	74-97-5	5.0	1.0	500
Bromodichloromethane	75-27-4	5.0	1.0	500
Bromoform (SPCC)	75-25-2	5.0	1.0	500
Bromomethane	74-83-9	5.0	1.0	500
2-Butanone	78-93-3	5.0	5.0	500
n-Butylbenzene	104-51-8	5.0	1.0	500
sec-Butylbenzene	135-98-8	5.0	1.0	500
tert-Butylbenzene	98-06-6	5.0	1.0	500
Carbon Disulfide	75-15-0	5.0	1.0	500
Carbon Tetrachloride	56-23-5	5.0	1.0	500
Chlorobenzene (SPCC)	108-90-7	5.0	1.0	500
Chloroethane	75-00-3	5.0	1.0	500
2-Chloroethyl Vinyl Ether	110-75-8	5.0	1.0	500
Chloroform (CCC)	67-66-3	5.0	1.0	500
Chloromethane (SPCC)	74-87-3	5.0	1.0	500
Chloroprene	126-99-8	5.0	1.0	500
2-Chlorotoluene	95-49-8	5.0	1.0	500
4-Chlorotoluene	106-43-4	5.0	1.0	500
1,2-Dibromo-3-chloropropane	96-12-8	5.0	1.0	500
Dibromochloromethane	124-48-1	5.0	1.0	500
1,2-Dibromoethane	106-93-4	5.0	1.0	500
Dibromomethane	74-95-3	5.0	1.0	500
1,2-Dichlorobenzene	95-50-1	5.0	1.0	500
1,3-Dichlorobenzene	541-73-1	5.0	1.0	500
1,4-Dichlorobenzene	106-46-7	5.0	1.0	500
cis-1,4-Dichloro-2-butene	1476-11-5	5.0	1.0	500
trans-1,4 Dichloro-2-butene	110-57-6	5.0	1.0	500
Dichlorodifluoromethane	75-71-8	5.0	1.0	500
1,1-Dichloroethane (SPCC)	75-34-3	5.0	1.0	500
1,2-Dichloroethane	107-06-2	5.0	1.0	500
1,1-Dichloroethene (CCC)	75-35-4	5.0	1.0	500
cis-1,2-Dichloroethene	156-59-2	5.0	1.0	500
trans-1,2-Dichloroethene	156-60-5	5.0	1.0	500
1,2-Dichloropropane (CCC)	78-87-5	5.0	1.0	500
1,3-Dichloropropane	142-28-9	5.0	1.0	500
2,2-Dichloropropane	594-20-7	5.0	1.0	500
1,1-Dichloropropene	563-58-6	5.0	1.0	500

Analyte	CAS No.	Reporting Limit[1]		
		Water or Soil µg/L or µg/Kg	Low Water µg/L	Methanol Extracts[2] µg/Kg
cis-1,3-Dichloropropene	10061-01-5	5.0	1.0	500
trans-1,3-Dichloropropene	10061-02-6	5.0	1.0	500
1,4-Dioxane	123-91-1	250	50	25,000
Ethyl Methacrylate	97-63-2	5.0	1.0	500
Ethylbenzene (CCC)	100-41-4	5.0	1.0	500
Freon TF	76-13-1	5.0	1.0	500
Hexachlorobutadiene	87-68-3	5.0	1.0	500
2-Hexanone	591-78-6	5.0	5.0	500
Isobutyl alcohol	78-83-1	250	50	25,000
Isopropylbenzene	98-82-8	5.0	1.0	500
4-Isopropyltoluene	99-87-6	5.0	1.0	500
Methacrylonitrile	126-98-7	5.0	1.0	500
Methyl Iodide	74-88-4	5.0	1.0	500
Methyl Methacrylate	80-62-6	5.0	1.0	500
4-Methyl-2-pentanone	108-10-1	5.0	5.0	500
Methyl-t-Butyl Ether	1634-04-4	5.0	1.0	500
Methylene Chloride	75-09-2	5.0	1.0	500
Naphthalene	91-20-3	5.0	1.0	500
Propionitrile	107-12-0	20	4.0	2,000
n-Propylbenzene	103-65-1	5.0	1.0	500
Styrene	100-42-5	5.0	1.0	500
1,1,1,2-Tetrachloroethane	630-20-6	5.0	1.0	500
1,1,2,2-Tetrachloroethane (SPCC)	79-34-5	5.0	1.0	500
Tetrachloroethene	127-18-4	5.0	1.0	500
Tetrahydrofuran	109-99-9	50	14	5,000
Toluene (CCC)	108-88-3	5.0	1.0	500
1,2,3-Trichlorobenzene	87-61-6	5.0	1.0	500
1,2,4-Trichlorobenzene	120-82-1	5.0	1.0	500
1,2,4-Trimethylbenzene	95-63-6	5.0	1.0	500
1,3,5-Trimethylbenzene	108-67-8	5.0	1.0	500
1,1,1-Trichloroethane	71-55-6	5.0	1.0	500
1,1,2-Trichloroethane	79-00-5	5.0	1.0	500
Trichloroethene	79-01-6	5.0	1.0	500
Trichlorofluoromethane	75-69-4	5.0	1.0	500
1,2,3-Trichloropropane	96-18-4	5.0	1.0	500
Vinyl Acetate	108-05-4	5.0	1.0	500
Vinyl Chloride (CCC)	75-01-4	5.0	1.0	500
Xylene (m,p)	1330-20-7	5.0	2.0	500
Xylene (o)	95-47-6	5.0	1.0	500

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.
[2] Methanol extracts 5 g to 10 mL.
CCC: Calibration Check Compound
SPCC: System Performance Check Compound

Table 2: BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0-40.0 percent of mass 95
75	30.0-60.0 percent of mass 95
95	Base peak, 100 percent relative abundance
96	5.0-9.0 percent of mass 95
173	Less than 2.0 percent of mass 174
174	>50.0 percent of mass 95
175	5.0-9.0 percent of mass 174
176	95.0-101.0 percent of mass 174
177	5.0-9.0 percent of mass 176

Table 3: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

Table 4: Control Limits[1,2] as Accuracy (%R) and Precision[3] (RPD)

Analyte	Low Level Water		Water, Medium Level Soils		Low Level Soils	
	%R	RPD	%R	RPD	%R	RPD
Acetone	80-135	30	60-135	30	45-165	30
Acrolein	65-150	40	20-205*	50*	70-145	35
Acrylonitrile	60-135	35	70-125	30	70-130	30
Allyl Chloride	70-125	30	75-125	30	75-120	30
Benzene	80-125	30	80-120	30	75-120	30
Bromobenzene	80-130	30	80-125	30	75-120	30
Bromochloromethane	80-125	30	80-120	30	75-120	30
Bromodichloromethane	85-130	30	80-135	30	85-130	30
Bromoform	**70-130**	30	**70-130**	30	75-120	30
Bromomethane	45-135	30	40-140	30	50-130	30
2-Butanone	80-145	30	75-135	30	60-160	30
n-Butylbenzene	75-140	30	75-140	30	80-125	30
sec-Butylbenzene	80-125	30	80-130	30	75-120	30
tert-Butylbenzene	80-140	30	75-135	30	75-120	30
Carbon Disulfide	75-120	30	80-125	30	75-120	30
Carbon Tetrachloride	75-120	30	75-120	30	75-120	30
Chlorobenzene	80-120	30	80-125	30	75-120	30
Chloroethane	85-135	30	**60-135**	30	75-120	30
2-Chloroethyl Vinyl Ether	80-125	30	80-125	30	10-220*	50*
Chloroform	80-125	30	75-125	30	75-120	30
Chloromethane	80-130	30	55-130	30	**50-130**	30
Chloroprene	75-120	30	65-140	40	75-120	30
2-Chlorotoluene	80-125	30	80-130	30	75-120	30
4-Chlorotoluene	80-130	30	75-130	30	80-125	30
1,2-Dibromo-3-chloropropane	70-140	30	**60-140**	30	55-125	30
Dibromochloromethane	70-125	30	75-130	30	75-120	30
1,2-Dibromoethane	**80-120**	30	**80-120**	30	75-120	30
Dibromomethane	85-130	30	85-125	30	75-120	30
1,2-Dichlorobenzene	**75-125**	30	**80-130**	30	75-120	30
1,3-Dichlorobenzene	80-130	30	80-130	30	80-125	30
1,4-Dichlorobenzene	85-130	30	**75-125**	30	75-120	30
cis-1,4-Dichloro-2-butene	75-130	30	70-130	30	55-130	30
trans-1,4-Dichloro-2-butene	75-130	30	65-140	35	65-130	30
Dichlorodifluoromethane	80-125	30	60-140	30	50-130	30
1,1-Dichloroethane	75-125	30	80-125	30	75-120	30
1,2-Dichloroethane	80-125	30	**70-130**	30	75-120	30
1,1-Dichloroethene	75-120	30	75-120	30	70-115	30
cis-1,2-Dichloroethene	75-120	30	80-125	30	75-120	30
trans-1,2-Dichloroethene	70-115	30	75-115	30	75-120	30
1,2-Dichloropropane	80-125	30	75-125	30	75-120	30
2,2-Dichloropropane	80-125	30	75-120	30	75-120	30
1,3-Dichloropropane	80-125	30	75-125	30	75-120	30
1,1-Dichloropropene	75-125	30	75-120	30	75-120	30
cis-1,3-Dichloropropene	80-125	30	75-130	30	75-120	30
trans-1,3-Dichloropropene	80-125	30	75-130	30	80-125	30
1,4-Dioxane	80-125	30	65-140	30	70-135	30

Analyte	Low Level Water		Water, Medium Level Soils		Low Level Soils	
Ethyl Methacrylate	80-125	30	75-130	30	75-120	30
Ethylbenzene	80-125	30	80-125	30	75-120	30
Freon TF	75-120	30	75-120	30	75-120	30
Hexachlorobutadiene	85-130	30	70-135	30	80-140	30
2-Hexanone	75-140	30	65-140	30	**45-145**	30
Isobutyl alcohol	70-135	30	55-145	45	70-125	30
Isopropylbenzene	80-130	30	80-130	30	75-120	30
4-Isopropyltoluene	80-125	30	80-130	30	80-125	30
Methacrylonitrile	80-125	30	70-130	30	70-130	30
Methyl Iodide	45-165*	50*	70-145	40	45-140	50
Methyl Methacrylate	75-125	30	75-120	30	70-125	30
4-Methyl-2-pentanone	85-130	30	80-125	30	75-125	30
Methyl-t-Butyl Ether	80-125	30	75-130	30	75-120	30
Methylene Chloride	75-120	30	75-120	30	70-115	30
Naphthalene	**70-155**	30	**55-140**	30	70-135	30
Propionitrile	75-120	30	75-120	30	65-130	30
n-Propylbenzene	80-130	30	80-130	30	75-120	30
Styrene	80-125	30	80-130	30	75-120	30
1,1,1,2-Tetrachloroethane	**80-130**	30	**80-130**	30	75-120	30
1,1,2,2-Tetrachloroethane	**70-135**	30	70-145	30	65-115	30
Tetrachloroethene	65-120	30	50-140	30	75-120	30
Tetrahydrofuran	85-130	30	80-125	30	70-125	30
Toluene	75-120	30	80-125	30	75-120	30
1,2,3-Trichlorobenzene	70-145	30	70-145	30	85-130	30
1,2,4-Trichlorobenzene	**70-140**	30	65-145	30	85-130	30
1,1,1-Trichloroethane	75-120	30	75-120	30	75-120	30
1,1,2-Trichloroethane	**75-130**	30	**75-125**	30	70-115	30
Trichloroethene	75-120	30	75-120	30	75-120	30
Trichlorofluoromethane	80-125	30	75-120	30	70-125	30
1,2,3-Trichloropropane	**75-125**	30	**75-125**	30	65-120	30
1,2,4-Trimethylbenzene	80-125	30	75-125	30	75-120	30
1,3,5-Trimethylbenzene	80-125	30	80-130	30	75-120	30
Vinyl Acetate	70-130	30	60-140	40	45-155	50
Vinyl Chloride	75-130	30	65-135	30	**60-125**	30
Xylene (m,p)	80-125	30	80-125	30	**80-125**	30
Xylene (o)	80-120	30	80-120	30	80-125	30
Surrogate Standards						
4-Bromofluorobenzene	**85-115**	NA	**85-115**	NA	**85-120**	NA
1,2-Dichlorobenzene-d4	80-125	NA	85-125	NA	80-125	NA
1,2-Dichloroethane-d4	80-125	NA	**70-120**	NA	80-125	NA
Toluene-d8	85-125	NA	85-120	NA	85-115	NA

[1] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.

[2] Those limits appearing in bold are limits for which the in-house limit it outside of the DoD required limit, and corrective action is taken based on the DoD limit per Section 13.2. However, no DoD limits were specifically calculated for Low level waters.

[3] RPD for MS/MSD only.

[*] Identified as poorly performing analyte. Decisions regarding batch acceptability are not based on analyte in the specific matrix indicated.

Table 5: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Minimum Frequency	Acceptance Criteria	Recommended Corrective Action[1]
BFB	Before initial and continuing calibration, every 12 hours	See Table 2	Reshoot, retune mass spectrometer
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CCCs: %RSD $\leq$ 30% SPCCs: mean RF per Section 10.4. Linear Regression: r $\geq$ 0.99 (0.995 for DoD) Quadratic: $r^2 \geq 0.99$	Correct problem and repeat initial calibration.
ICV	After each initial calibration	%Difference ± 25%	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Beginning of each 12-hour window, as established by a compliant BFB.	SPCCs: must meet minimum RF CCCs: %D ≤ 20%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs at different concentrations pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and samples are non-detects.
MB	One per batch of 20 or fewer samples	< RL DoD: $\leq$ ½ RL for all analytes except < RL for acetone, 2-butanone, and methylene chloride for any sample $\geq$ RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB and samples (if samples have been run), and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, reanalysis may not be required.
LCS	One per batch of 20 or fewer samples	Evaluated against control limits in Table 4, 4 Marginal Exceedances allowed.	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS and samples (if samples have been run), and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch, DoD: project specific per extraction batch SD: Per client request	Evaluated against control limits in Table 4	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze. Flag all reported values outside of control limits.
Surrogate Standard	All field and QC samples	Evaluated against control limits in Table 4	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.
Internal Standard	All field and QC samples	Area between 50-200% of area of daily calibration internal standard area	Same as above.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that data quality is known and documented. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Appendix A: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP LP-LB-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

All standards are prepared using volumetric glassware including Hamilton syringes. All standards are made with purge and trap grade methanol, demonstrated to be analyte free and standards are stored at -10 to -20^oC in amber glass mini-inert vials, except for the routine level water and medium level surrogate and internal standard solutions, which may be stored in volumetric flasks at 2-6^oC.

Table Legend:
C_P = Concentration of Parent Standard
V_P = Volume of Parent Standard
V_S = Volume of Prepared Standard
C_S = Theoretical Concentration of Prepared Standard

TUNING STANDARD

Bromofluorobenzene – 25 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene	30003	5000 μg/mL	125 μL	25 mL	25 mg/L

Expiration Date: 6 months from preparation or expiration of parent, whichever is earlier.

CALIBRATION STANDARDS

Internal Standard Spiking Solution – 50 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Chlorobenzene-d_5 1,4-Dichlorobenzene-d_4 Fluorobenzene	50684	1000 μg/mL	1250 μL	25 mL	50 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

Surrogate Standard – 50 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene 1,2-Dichlorobenzene-d_4 1,2-Dichloroethane-d_4 Toluene-d_8	53837	2000 μg/mL	625 μL	25 mL	50 mg/L

Restek Catalog #
Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

8260 Calibration – Mixed Concentration

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene 1,2-Dichlorobenzene-d_4 1,2-Dichloroethane-d_4 Toluene-d_8	Surrogate: 53837	2000 μg/mL	200 μL	4 mL	100 mg/L
Vinyl Acetate	30216	2000 μg/mL	200 μL	4 mL	100 mg/L
Methyl acrylate Methyl methacrylate Allyl chloride Nitrobenzene Acrylonitrile Pentachloroethane Ethyl methacrylate	Mix 7B: 30202B	2000 μg/mL	200 μL	4 mL	100 mg/L
54 volatile Components, see Catalog	Mega-mix: 30431	2000 μg/mL	200 μL	4 mL	100 mg/L
1,4-Dioxane Isobutyl alcohol Tetrahydrofuran Propionitrile trans-1,4-Dichloro-2-butene 1,1,2-Trichlorotrifluoroethane 2-Chloro-1,3-butadiene Carbon disulfide Methacrylonitrile Methyl-tert-butyl ether Iodomethane cis-1,4-Dichloro-2-butene	Custom: 56531	100,000 μg/mL 100,000 μg/mL 18,000 μg/mL 8000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL	200 μL	4 mL	5000 mg/L 5000 mg/L 900 mg/L 400 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L

Expiration Date: 2 months from preparation or expiration of parent, whichever is earlier.

8260 Calibration Gas – 100 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromomethane Chloroethane Chloromethane Dichlorodifluoromethane Trichlorofluoromethane Vinyl Chloride	30042	2000 μg/mL	75 μL	1500 μL	100 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

8260 Calibration Added – 100 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
2-Chloroethylvinylether	30265	2000 μg/mL	200 μL	4 mL	100 mg/L
Acrolein	53547	5000 μg/mL	80 μL	4 mL	100 mg/L
Acetone 4-Methyl-2-pentanone Methyl ethyl ketone Tetrahydrofuran 2-Hexanone	Mix 7A: 30202A	2000 μg/mL	200 μL	4 mL	100 mg/L

Expiration Date: 2 months from preparation or expiration of parent, whichever is earlier.

LOW LEVEL WATER CALIBRATION

Internal Standard Low – 25 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Chlorobenzene-d_5 1,4-Dichlorobenzene-d_4 Fluorobenzene	50684	1000 μg/mL	150 μL	6 mL	25 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

Surrogate Standard Low – 25 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene 1,2-Dichlorobenzene-d_4 1,2-Dichloroethane-d_4 Toluene-d_8	53837	2000 μg/mL	75 μL	6 mL	25 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

8260 Calibration Low – Mixed Concentration

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene 1,2-Dichlorobenzene-d_4 1,2-Dichloroethane-d_4 Toluene-d_8	Surrogate: 53837	2000 μg/mL	55 μL	4.4 mL	25 mg/L
Vinyl Acetate	30216	2000 μg/mL	55 μL	4.4 mL	25 mg/L
Methyl acrylate Methyl methacrylate Allyl chloride Nitrobenzene Acrylonitrile Pentachloroethane Ethyl methacrylate	Mix 7B: 30202B	2000 μg/mL	55 μL	4.4 mL	25 mg/L
54 volatile Components, see Catalog	Mega Mix: 30431	2000 μg/mL	55 μL	4.4 mL	25 mg/L
1,4-Dioxane Isobutyl alcohol Tetrahydrofuran Propionitrile trans-1,4-Dichloro-2-butene 1,1,2-Trichlorotrifluoroethane 2-Chloro-1,3-butadiene Carbon disulfide Methacrylonitrile Methyl-tert-butyl ether Iodomethane cis-1,4-Dichloro-2-butene	Custom: 56531	100,000 μg/mL 100,000 μg/mL 18,000 μg/mL 8000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL	55 μL	4.4 mL	1250 mg/L 1250 mg/L 225 mg/L 100 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L

Expiration Date: 2 months from preparation or expiration of parent, whichever is earlier.

8260 Calibration Gas Low – 25 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromomethane Chloroethane Chloromethane Dichlorodifluoromethane Trichlorofluoromethane Vinyl Chloride	30042	2000 μg/mL	19 μL	1520 μL	25 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

8260 Calibration Added Low – Mixed Concentration

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
2-Chloroethylvinylether	30265	2000 μg/mL	55 μL	4.4 mL	25 mg/L
Acrolein	53547	5000 μg/mL	110 μL	4.4 mL	125 mg/L
Acetone 4-Methyl-2-pentanone Methyl ethyl ketone Tetrahydrofuran 2-Hexanone	30202A	2000 μg/mL	275 μL	4.4 mL	125 mg/L

Expiration Date: 2 months from preparation or expiration of parent, whichever is earlier.

Appendix B: Equations

Response Factor (RFx) = $$\frac{Area_x \text{ X } Concentration_{is}}{Area_{is} \text{ X } Concentration_x}$$

Where: x=compound, is = Internal Standard

Relative Retention Time (RRT) = $$\frac{Retention\ Time_x}{Retention\ Time_{is}}$$

Where: x=compound, is = Internal Standard

Mean Response Factor ($\overline{RF}$) = $$\frac{\sum_{i=1}^{n} RF_i}{n}$$

where: n = number of calibration levels

Standard Deviation of the Response Factor (SD) = $$\sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)2}{n-1}}$$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Response = $$\frac{SD}{\overline{\overline{RF}}} \times 100\%$$

Percent Difference (%D) = $$\frac{RF_v - \overline{RF}}{\overline{\overline{RF}}} \times 100\%$$

where: RF_v = Response Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $$\frac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}} \text{ X } 100\%$$

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Water

$$C_x = \frac{A_x \times C_{is}}{A_{is} \text{ X Mean RF}} \times DF$$

Solids

$$C_x = \frac{A_x \times C_{is}}{A_{is} \text{ X Mean RF x Percent Solids}} \times DF$$

Where

C_x = Concentration of compound (µg/L)
A_{is} = Area of quantification ion for associated internal standard.
A_x = Area of quantification ion for compound.
C_{is} = Concentration of associated internal standard (µg/L).
DF = Dilution Factor.
Mean RF = Mean Response Factor from initial calibration, or 1 for a tentatively identified compound

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Appendix C: Terms & Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed.

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Calibration Check Compounds (CCCs): Selective analytes from the compound list that are used to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Internal Standard: a known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery for each analyte.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

System Performance Check Compounds (SPCCs): Selective analytes from the compound list that are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.

STANDARD OPERATING PROCEDURES FOR THE CLOSED-SYSTEM PURGE AND TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES

Approvals and Signatures

Laboratory Director:	Michael F. Wheeler, PhD.	Date:	5/12/03
QA Manager:	Kirstin L. McCracken	Date:	5-9-03
GC/MS Manager:	Bryce E. Stearns	Date:	5/9/03

1.0 SCOPE AND APPLICATION

1.1 This SOP is describes the laboratory procedure for a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in soils, sediments, and solid waste.

1.2 The closed-system purge and trap method utilizes a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. This procedure is applicable to low-concentration soils in the range of 0.5-200ug/kg.

1.3 The sample preparation procedures for high concentration soil and oily wastes are included because the use of this procedure is based on the results of screen data and/or sample history. Soil and waste samples that approximate concentrations above 200ug/kg are prepared according to this SOP and then purged following the laboratory SOP for purge and trap of aqueous samples (SW-846 Method 5030B).

2.0 SUMMARY OF METHOD

2.1 Low concentration samples (0.5-200ug/kg) for volatile organic compounds (VOCs) are determined by collecting an approximately 5g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum sealed screw cap that already contains a stirring bar and a sodium bisulfate preservative solution or Encore™ sampling device. The samples are shipped to the laboratory and then placed, unopened, into the instrument carousel. Samples in Encore™ sampling device are transferred to sample vials within 48 hours of receipt. Immediately before analysis, organic-free reagent water, surrogates, and internal standards are automatically added without opening the sample vial. The vial containing the sample is heated to 40°C and the volatiles are purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and back flushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 This procedure is based on SW-846 Method 5035.

3.0 DEFINITIONS

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Matrix: the substrate of a test sample.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is +100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Quality Control Sample: a control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

4.0 INTERFERENCES

4.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.3 The laboratory where volatile analysis is performed should be completely free of solvents. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

5.0 SAFETY

5.1.1 The toxicity or carcinogenity of each chemical used in this procedure has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be minimized as reasonably possible. A reference file of Material Safety Data Sheets (MSDS) for this test method is available to all personnel and must be read prior to performing this procedure.

5.2 All laboratory personnel must be familiar with the laboratory environmental health and safety plan described in the STL Corporate Safety Manual (CSM).

6.0 EQUIPMENT AND SUPPLIES

6.1 Sample Containers

44mL screw cap vials that can be sealed with a PTFE-faced silicon septa

6.2 Purge and Trap System

Varian Chromatography Systems Archon Purge-and-Trap Tekmar ALS 2050, Tekmar AQUATEK 50 or equivalent

Purge & Trap: Tekmar LSC 2000; VOCARB 3000 trap or equivalent

6.3 Syringes

250 μL-5mL gas-tight hypodermic syringes with Luer-Lok tip

Micro syringe 10-100μL

6.4 Miscellaneous

Teflon Coated Stir Bars

Stainless Steel Spatula

Encore™ T-Bar

Top loading balance capable of weighing 0.01g.

7.0 REAGENTS AND STANDARDS

7.1 Reagent water-deionized water that has been filtered through the laboratory's Milli Q plus ™ filtration system, boiled for one hour and purged with helium for a minimum of fifteen minutes. The reagent water must be stored in clean, narrow-mouth bottles with Teflon lined septa and screw caps.

7.2 Methanol- Purge and Trap quality or equivalent

7.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent

7.4 Standards

Internal standard and surrogate solutions are prepared in the laboratory from certified, stock standards purchased from commercial vendors by diluting a volume of stock standard solution in methanol. The procedure and recommended concentration for calibration standards is given in the laboratory SOP for the determinative method to be used in conjunction with this SOP.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 SW-846 Method 5035 describes several sampling and preservation options for both low and high-level VOA analysis. The preparation of vials used during sample collection is dependent on the concentration range of the sample. The sample collection procedure should be described and performed in accordance with the client's sampling plan following the guidance of SW-846 Method 5035. At this time, the laboratory does not perform sample collection, thus these procedures are not applicable to this SOP. The laboratory may provide on client request, prepared, certified vials and Encore™ sampling devices, which are shipped directly to the sampling site. The procedures for vial preparation are described in section 11.0 of this SOP.

8.2 Samples should be preserved in the field and collected in triplicate to ensure sufficient sample is available for reanalysis. A sufficient amount of sample to fill a 125mL jar should also be collected, unpreserved as the "bulk" sample. The "bulk" sample is used for dry-weight determination, screening and high concentration analysis, if necessary. Samples expected to be low concentration, should be preserved with sodium bisulfate. High concentration samples should be preserved with methanol.

8.3 Immediately following collection, samples should be cooled to 4°C (± 2°C) and maintained at that temperature until the time of analysis.

8.4 The holding time is 14 days from collection. Samples that were collected in the Encore™ device must be transferred into soil sample vials as soon as possible after collection or analyzed within 48 hours. Other holding times may be specified as required by the CLP SOW, client sampling plan or other program requirements.

8.5 Samples stored from the time of receipt in the laboratory until 60 days after delivery of the reconciled data package report. Unless otherwise specified by a federal, state or client-specific protocol, samples are disposed of after 60 days in a manner that complies with all applicable regulations.

9.0 QUALITY CONTROL

9.1 The procedures for quality control are given in laboratory SOPs for the determinative methods used in conjunction with this procedure.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prior to using the closed system purge and trap procedure for any GC or GC/MS method, the instrument must be calibrated. The procedures for calibration are given in the laboratory SOP for the determinative method along with the instrument operating conditions and quality control requirements including standard preparation.

11.0 PROCEDURE

11.1 Preparation of Sample Vials

The laboratory purchases pre-made, pre-certified preserved vials from a commercial vendor. Vial preparation procedures are included in this section in the event that the laboratory needs to prepare the vials in-house when there is an insufficient quantity vials commercially available.

<u>Low Concentration Sample Vials (Preserved)</u>

Place a clean magnetic stir bar in a 44mL sample vial.

Add 5mL of 20% sodium bisulfate solution.

Seal and cap the vial with a screw cap and septum seal. Affix a label.

Place the vial on a calibrated top-loading balance and record the tare weight on the label and in the logbook designated for this purpose.

<u>High Concentration Sample Vials (Preserved)</u>

Add 10mL of methanol to a 44mL sample vial.

Seal and cap the vial with a screw cap and septum seal. Affix a label.

Place the vial on a calibrated top-loading balance and record the tare weight on the label and in the logbook designated for this purpose.

11.2 Preparation of Encore™ Samples

<u>Low Concentration Samples</u>

Within 48 hours of sample receipt, transfer the contents of the Encore™ sampling device into a pre-cleaned 40mL sample vial to which a Teflon coated stir-bar and 5mL of sodium bisulfate solution has been added Cap and seal the vial with the PFTE lined septum seal. Place the vial on a calibrated top-loading balance and record the final weight in the logbook designated for this purpose. Duplicate sample preparations require the assignment of a unique aliquot identifier written on the label (i.e. STL L# M1,M2,M3, etc) as necessary.

Note: Samples that effervesce when placed in the sodium bisulfate solution should be prepared in 40mL sample vials containing a Teflon coated stir bar and 5mL of VOA free lab water. These samples should be stored frozen until the time of analysis.

Medium Concentration Samples

Within 48 hours of sample receipt, transfer the contents of the Encore™ sampling device into a pre-cleaned 40mL sample containing 10mL of purge and trap grade Methanol (medium level concentration samples). Cap and seal the vial with the PFTE lined septum seal. Place the vial on a calibrated top-loading balance and record the final weight in the logbook designated for this purpose. Duplicate Methanol preserved sample preparations require the assignment of a unique aliquot identifier written on the label (i.e. STL L# N1,N2,N3, etc) as necessary.

11.3 Sample Screening

Screen all samples prior to analysis following the procedures in laboratory SOP LM-MV-3810, *VOA Screen for Water, Soil, and Waste.* Use the screen data to determine which sample preparation procedure is appropriate.

If the approximate concentration range is 0.5-200ug/kg, proceed to the section 11.4.

If the approximate concentration range is above 200ug/kg or if the sample is an oily waste, proceed to section 11.5.

11.4 Closed System Purge and Trap (Low Concentration Samples)

Condition the purge and trap device by initiating the bake cycle.

Obtain the samples from sample management. Inspect the sample vial to ensure that it is hermetically sealed and intact.

Place the vial on a calibrated top-loading balance and record the weight in the logbook designated for this purpose. If any soil is visible on the exterior of the vial or cap, carefully remove prior to taking the weight measurement.

Allow the sample to warm to room temperature. Shake the vial gently to ensure that the contents move freely and that stirring will be effective.

Prepare a vial each for the MB and LCS by transferring 5mL sodium bisulfate into a 44mL vial.

Prepare vials each for each calibration standard by transferring the appropriate volume of calibration standard solution into a 44mL vial to which reagent water has been added. The recommended concentration for each calibration level is given in the SOP for the associated determinative method.

Add spike solution to the LCS and any sample(s) selected for the MS/MSD.

Inject internal standard and surrogate solution to every sample, QC item, and calibration standard through the septum.

Place the vials in the autosampler. Program the autosampler to heat the sample at 40°C for 1.0 minute prior to beginning the purge process.

Purge the sample with helium gas for 11.0 minutes at temperature of 40°C while agitating the sample with the magnetic stir bar. After the sample has purged, select the desorb mode and preheat the trap to 240°C without a flow of desorption gas. Begin the flow of desorption gas at a rate of ~10mL/minute and simultaneously begin the temperature and data acquisition program. While the trapped components are being introduced into the gas chromatograph, the automated sampling system drains and rinses the purge vessel twice during the sample desorption step.

After desorbing the sample for four minutes, recondition the trap with the bake cycle before returning the purge and trap system to the purge mode. Maintain the trap temperature at 260°C for approximately seven minutes. Turn off the heater and halt the purge flow through the trap. When the trap is cool, analyze the next sample.

Perform qualitative and quantitative analysis following the appropriate laboratory SOP for the determinative method requested.

11.5 High Concentration and Oily Waste Samples

High concentration samples (above 200ug/kg) and oily waste samples are solvent extracted or diluted and introduced to the GC or GC/MS using the purge and trap method for aqueous samples described in laboratory SOP LM-MV-5030B. This section describes the procedures used to prepare the soil sample for the aqueous purge and trap method.

<u>High Concentration Soil Samples (Unpreserved)</u>

Using a top loading balance measure 5g of sample into a 44mL vial, to which 10mL of methanol has been added. Quickly reseal the vial to minimize the loss of volatiles. Record the weight in the logbook designated for this purpose. Shake the vial for two minutes.

Partially fill a 44mL vial with reagent water. Transfer 880uL of the extract into the vial using a syringe or calibrated pipette. Adjust the volume using reagent water.

Inject internal standard and surrogate solution to each sample through the septum.

Analyze following the laboratory SOP for purge and trap of aqueous samples in conjunction with the determinative method.

High Concentration Soil Samples (Preserved)

Place the vial on a calibrated top-loading balance and record the weight in the logbook designated for this purpose.

Partially fill a 44mL vial with reagent water. Transfer 880uL of the extract into the vial using a syringe or calibrated pipette. Adjust the volume using reagent water.

Inject internal standard and surrogate solution to each sample through the septum.

Analyze following the laboratory SOP for purge and trap of aqueous samples in conjunction with the determinative method.

Oily Waste Samples (Unpreserved)

Using a top loading balance measure 1g of sample into a 44mL vial, to which 10mL of methanol has been added. Quickly reseal the vial to minimize the loss of volatiles. Record the weight in the logbook designated for this purpose. Shake the vial for two minutes.

Partially fill a 44mL vial with reagent water. Transfer 880uL of the extract into the vial using a syringe or calibrated pipette. Adjust the volume using reagent water.

Inject internal standard and surrogate solution to each sample through the septum.

Analyze following the laboratory SOP for purge and trap of aqueous samples in conjunction with the determinative method.

12.0 CALCULATIONS

12.1 This section is not applicable to this procedure.

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Primary review is performed by the analyst(s) that performed the procedure. The data undergoes secondary review by a senior data review analyst. Problems encountered during analysis are documented and reported in the case narrative provided with the data package report.

14.0 METHOD PERFORMANCE

14.1 An Initial Demonstration of Capability is required for each analyst before unsupervised performance of this method.

14.2 An Initial Method Detection Limit (MDL) determination for each test method referenced in this SOP is performed following the procedure described in the reference method, 40CFR, Part 136, Appendix B and laboratory SOP LP-LB-009. The MDL is verified or repeated when a significant change to the method occurs. Significant changes include the use of alternate reagents or standard reference materials, new instrumentation or the use of alternate sample preparation procedures.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 The laboratory optimizes technology to minimize pollution and reduce the production of hazardous waste whenever possible.

15.2 The laboratory procedures for waste management comply with applicable federal, state and local regulations and are described in SOP LP-LB-001HAZWD.

16.0 REFERENCES

16.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846),* Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996

16.2 *Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, (Current SOW),* USEPA Contract Laboratory Program.

17.0 TABLES, DIAGRAMS & FLOWCHARTS

17.1 This section is not applicable to this procedure.

STANDARD OPERATING PROCEDURE
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS
SW-846 Method 8270C
Matrix: Non-Potable Water, Solid & Chemical Materials, Tissue

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APPROVAL SIGNATURES

Laboratory Director:	Christopher A. Ouellette	Date: December 12, 2005
QA Manager:	Kirstin L. McCracken	Date: December 12, 2005
Organics Manager:	Jennifer L. Clements	Date: December 12, 2005

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the laboratory procedure used to determine the concentration of semivolatile organic compounds in extracts derived from non-potable water, solid and chemical materials, and tissue. This SOP is applicable to the analytical procedure only; the extraction and extract cleanup methods referenced in this SOP are described in the following laboratory SOPs:

LM-OP-3550	Ultrasonic Extraction
LM-OP-3540	Soxhlet Extraction
LM-OP-3510	Separatory Funnel Extraction
LM-OP-GPC	Gel Permeation Chromatography (GPC)
LP-OP-3541	Automated Soxtherm Extraction
LM-OP-3580A	Waste Dilution

1.2 The analytes that can be determined by this procedure and their associated Reporting Limits (RL) are listed in Table 1A & 1B, Section 18.

1.3 The following problems have been associated with compounds analyzed by this method: dichlorobenzidine and 4-chloroaniline may be subject to oxidative losses during solvent concentration; hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reactions in acetone solution, and photochemical decomposition; and n-nitrosodiphenylamine decomposes in the gas chromatograph inlet forming diphenylamine and, consequently, is detected as diphenylamine. Likewise, 1,2-Diphenylhydrazine is detected as Azobenzene due to decomposition during the GC/MS analysis.

2.0 SUMMARY OF METHOD

2.1 Samples are prepared for analysis by GC/MS using an appropriate extraction method and if necessary, extract cleanup procedure. An aliquot of extract is injected into the gas chromatograph (GC), where it is volatilized in the injection port and swept onto the chromatographic column in which a temperature program is used to separate the target compounds, and they are then detected by a mass spectrometer (MS). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.2 This procedure is based on SW-846 Method 8270C, Revision 3, December 1996.

3.0 DEFINITIONS

A list of terms and definitions is given in Appendix B.

4.0 INTERFERENCES

4.1 Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the extracted ion current profiles (EICPs). All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

4.2 Injection syringes should be adequately flushed with solvent between injections in order to remove all traces of the prior sample.

4.3 Co-extracted Interferences may include lipids, polymers, copolymers, proteins, natural resins, cellular components, viruses, steroids, and high-molecular weight compounds. GPC, which is size exclusion chromatography, is appropriate for cleanup of these types of polar and non-polar interferences.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 Primary Materials Used

Table 2, Section 18 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

- Methylene chloride

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance: Capable of weighing to 0.1 mg.

6.2 Containers

- 2 mL Autosampler vials with 200 uL inserts, PFTE crimp top.
- 4 mL sample vials with PFTE lined screw top caps.

6.3 Computer Hardware/Software: GCMS Acquisition Platform - Hewlett-Packard ChemStations. Data Processing - Hewlett-Packard 9000-series computers, an HP9000 D250 (Chemsvr4) and an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5.

6.4 Instrumentation

- SVOA Autosampler: HP 7673A™, CTC A200S™ or equivalent.
- Gas Chromatograph: Hewlett-Packard™ 5890 GC, 6890 GC or equivalent.
- Mass Spectrometer: Hewlett-Packard™ 5971, 5972and 5973 MSD or equivalent.
- Primary Column: Restek™ RTX-5 30m x 0.25mm ID x .25 um film thickness or equivalent.
- Guard Column: Restek™ Deactivated 5m x 0.25 mm ID or equivalent.
- Column unions: Restek Press-Tights™ or equivalent.
- Injection port liners: Single Goose Neck, borosilicate glass. Restek™ 20799 or equivalent.
- Injection port septa: HP™, 11 mm Thermo Red or equivalent.
- Data System: Hewlett-Packard Chem server™, Target 3.5 processing software and Hewlett-Packard ChemStation software for instrument control and acquisition.

6.5 Syringes: 10 uL, 25 uL, 50 uL, 100 uL, 1000 uL.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Methylene Chloride (CH_2Cl_2), Pesticide quality.

7.2 Standards

Stock standard solutions are purchased from commercial vendors and stored according to manufacturer instructions. The stock standard solutions remain unopened until time of use and are considered acceptable until the expiration date given by the manufacturer. Intermediate and working standards are prepared in the laboratory by dissolving a volume of stock standard solution in an appropriate solvent and diluting to a specified volume. Standard solutions are stored in amber glass vials with Teflon lined screw caps at a temperature of 4^{o}C (± 2). Unless otherwise noted, prepared standard solutions are assigned an expiration date of 6 months from date of preparation unless the expiration date of the parent standard expires sooner in which case, the expiration date of the parent standard is used.

Calibration Mix (CAL MIX): Dilute appropriate volumes of commercially purchased stock standard solutions in methylene chloride to achieve a final concentration of 166.67 ng/uL for each analyte and surrogate compound.

Working Calibration Standards: Dilute an appropriate volume(s) of the CAL MIX in methylene chloride to achieve a series of standards at the following concentrations: 10, 25, 40, 60, and 80 ng/uL (20, 50, 80, 120, and 160 ng per 2-ul injection)

Internal Standard Solution: Dilute an appropriate volume of the commercially prepared stock standard solution (i.e. Restek™ Internal Standard Mix) in methylene chloride to achieve a concentration of 500 ng/uL.

Initial Calibration Verification (ICV) (25ng/uL): Dilute appropriate volumes of second source stock standard solutions in methylene chloride to achieve a final concentration of 25 ng/uL.

Decafluorotriphenylphosphine (DFTPP) Mix (25ng/uL): Dilute appropriate volumes of commercially prepared stock standard solutions (DFTPP, Benzidine, Pentachlorophenol, and DDT) in methylene chloride to achieve a final concentration of 25 ng/uL.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample extracts must be stored at 4°C ± 2° until the time of analysis. The analytical holding time is 40 days from date of sample extraction.

8.2 Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 The minimum frequency requirements, acceptance criteria and recommended corrective action for all QC samples are summarized in Section 18, Table 4. Below is a summary of each type of QC sample that is analyzed with the method.

9.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each batch of 20 or fewer samples. These samples show that the laboratory is in control independent of the sample matrix.

9.3 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each batch of 20 samples. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.4 Surrogate standards are added to all field and QC samples before preparation and/or analysis to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.5 Internal Standards – All samples are spiked with internal standards as described in Section 7.

9.6 Instrumental QC standards include a DFTPP every 12 hours, before each Initial Calibration (ICAL) and CCV. A five-point ICAL is generated for each target analyte. After the ICAL, an ICV, also referred to as a second source standard, is analyzed to verify the ICAL standard formulation. Continuing Calibration Verification (CCV) standards are analyzed every 12 hours immediately following the DFTPP.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prior to the acquisition of a calibration curve, CCV, or the analysis of samples, a 2 μL aliquot of DFTPP (25 μg/mL) is injected into the GC. The data processing system acquires and averages three scans (apex scan, scan prior, and scan preceding) and performs background subtraction of the single scan prior to the elution of the DFTPP. The DFTPP must meet the criteria in Table 4 before initial or continuing calibration may proceed. If criteria are not met, retune the instrument. Analysis may not proceed until tune criteria are met.

The DFTPP is also used to evaluate column performance and injection port inertness. The Tailing factor for Benzidine and pentachlorophenol are evaluated and must be less than 5.0 and 3.0, respectively. For DoD projects, DDT breakdown is evaluated. The degradation of DDT must not exceed 20%. If these criteria are not met, perform corrective action before further analysis. Corrective action may include cleaning of the injection port or clipping the column.

10.2 Samples may be run for 12 hours after successful initial and/or continuing calibration. The official start time of the 12-hour window is the time of the DFTPP injection. The last sample in the window must be injected within 12 hours of that time.

10.3 Initial Calibration (ICAL)

Add 4-uL of internal standard solution to each calibration standard to achieve a final internal standard concentration of 20-ng/uL in extract (40-ng on column, 2-uL injection). Inject 2-uL of each calibration standard onto the GC/MS system. Ensure the analysis of the calibration standards is within the 12-hour analytical window that was started at the time of injection of the DFTPP standard.

Calibrate the instrument with a minimum of five calibration standards that include all compounds of interest at each concentration level. The recommended concentration for each calibration level is 10, 25, 40, 60, and 80 ng/uL (20, 50, 80, 120, and 160 ng per 2-ul injection). The 10-ng/uL standard is not used for the following analytes: Benzoic acid, 2,4,5-Trichlorophenol, 2-Nitroaniline, 3-Nitroaniline, 2,4-Dinitrophenol, 4-Nitrophenol, 4-

Nitroaniline, 4,6-Dinitro-2-methylphenol, Pentachlorophenol, and Benzidine. To provide a five-point calibration for these compounds, a 180-ng/uL standard is analyzed. While all analytes are present in the 180-ng/uL, it only needs to be processed for those above named analytes.

If samples are to be analyzed for the extended 8270 Appendix IX target compound list (See Table 1B), use a second set of calibration standards to minimize constituent deterioration due to compound interaction. Analyze two sets each of five calibrations standards (ten total) at the concentrations listed above. Each calibration set includes specific compounds that are determined to be free from interactions of interference.

10.4 For each target analyte and surrogate, calculate the mean Response Factor (RF) from analyses of the five calibration solutions. Calculate the standard deviation (SD) and Percent Relative Standard Deviation (%RSD - see Appendix A for calculations).

The following criteria must be met for a calibration to be considered acceptable:

- System Performance Check Compounds (SPCCs) must meet the following minimum mean RF: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol – 0.050.
- Calibration Check Compounds (CCCs) must have a %RSD of $\leq$ 30%: Acenaphthene, 4-Chloro-3-methylphenol, 1,4-Dichlorobenzene, 2,4-Dichlorophenol, Hexachlorobutadiene, 2-Nitrophenol, N-nitrosodiphenylamine, Phenol, Di-n-octyl phthalate Pentachlorophenol, Fluoranthene, 2,4,6-Trichlorophenol, Benzo(a)pyrene. If the CCCs are not included in the list of analytes, then the criteria in 10.5 should be used.
- The Relative Retention Time (RRT) for each target analyte in each calibration standard should agree within 0.06 RRT units (See Appendix A for calculation).

10.5 If the %RSD $\leq$ 15%, for all analytes, the calibration is acceptable and the mean RF may be used for quantification. If this criterion is not met, either use another suitable quantification method, or correct the problem and repeat the calibration.

10.6 Alternate Quantification. In some cases, it may be preferable to use either linear regression or Quadratic Equations to quantify the compounds. The following approaches may be used:

Linear Regression - A curve of concentration vs. peak area is generated for each analyte and the correlation coefficient is calculated. The calibration must have a correlation coefficient (r) $\geq$ 0.99 (0.995 for DoD) for acquisition of samples to continue. The use of linear regression requires a minimum of 5 calibration points. See SW-846 Method 8000B for linear regression calculations.

Quadratic Equation - For some compounds, the response is not linear, and in this case a quadratic equation may be employed. For those compounds, the coefficient of determination (r^2) is generated. The coefficient of determination must be $\geq$ 0.99 for acquisition to continue. The uses of quadratic equations require a minimum of 6 calibration points for second order regression and 7 points for third order regression. See SW-846 Method 8000B for quadratic equation calculations.

Once a method of calibration is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is generated.

10.7 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV. To prepare the ICV, add 4-uL of internal standard solution to 100-uL of the 25-ng/uL ICV solution, and inject 2-uL of the solution to achieve an on-column concentration of 50-ng. The calculated concentration of each analyte must be within ± 25% of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate.

If after successful analysis of the ICV time remains in the 12 -hour analytical window, samples may be analyzed without analysis of a continuing calibration verification check standard (CCV); otherwise a CCV must be performed.

10.8 Continuing Calibration Verification (CCV)

At the beginning of each 12-hour shift in which samples are to be run, analyze a DFTPP as outlined in Section 10.1. A CCV standard, at or below mid-calibration range, is analyzed at the beginning of each 12-hr work shift. The concentration of the CCV is varied.

Inject 2 uL of the prepared CCV standard, acquire the data and evaluate the results. If samples are to be analyzed for the extended Appendix IX target compound list (See Table 1B), analyze 2 CCVs containing the same target compounds as established in the initial calibration.

Calculate the RF and percent difference or drift (see Appendix A for calculation) for each target analyte and surrogate standard. The SPCCs must meet the criteria outlined in Section 10.4. The percent difference or drift for the CCCs must be ≤ 20%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

In addition, the internal standard retention time should not change by more than 30 seconds from the retention time in the mid-point standard of the most recent ICAL. The EICP area of the internal standards in the calibration verification standard should not change by more than a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent ICAL.

If the CCV fails, it may be repeated once. If it still fails, corrective action must be taken. The analysis may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples analyzed after a failing CCV must be reanalyzed, unless the analyte in the CCV is high and that analyte is not detected in the associated samples.

10.9 Troubleshooting: the following items can be checked in case of calibration, QC or instrument failures:

- Benzidine is subject to oxidative loss during extraction and chromatographs poorly, injection port and/or column maintenance may be required.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet, injection port and/or column maintenance may be required.
- N-nitrosodiphenylamine decomposes in the inlet and cannot be separated from diphenylamine. Compound is reported as N-nitrosodiphenylamine.
- 1,2-Diphenylhydrazine decomposes to Azobenzene in the analytical portion of the procedure and as such is reported as Azobenzene.
- Acid compounds are subject erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material, injection port and/or column maintenance may be required.
- Loss of sensitivity for higher boiling compounds and internal standards may be indicative of a leak at the inlet. Replace septa and/or re-tighten lower inlet connection.
- Carryover contamination may indicate empty rinse vials.

11.0 PROCEDURE

11.1 Extract Preparation & Analysis

Allow the extracts to warm to room temperature. Transfer 100-uL of extract to a 1-mL auto-sampler vial with insert. Add 4-uL of the internal standard solution to each vial and seal the vial with a PTFE lined crimp top cap. If an alternate extract volume is used (e.g. 50 uL extract) adjust the volume of internal standard proportionately.

When extract screening has been performed, prepare sample dilutions by diluting an appropriate volume of sample extract in methylene chloride. Serial dilutions may be required if the relative volumes needed for a single dilution step exceed the accuracy of the pipettes. For example, a sample requires a 0.1% analysis in order to have target constituents within the upper half of the calibrated range. This level of dilution in a 100-uL aliquot requires 0.1-uL of sample extract. However, the gradations of the microliter pipette are to 0.2-uL. In this situation, it is necessary to perform a serial dilution of 1:100 (1.0%) and a 10:100 (10%) to achieve an analysis concentration of 0.1% of the original extract.

11.2 Instrument Operating Conditions

Gas Chromatography/Mass Spectrometry - Data is acquired and stored over the nominal mass range of 35-500 atomic mass units (amu) with a total cycle time (including scan overhead time) of one second per scan at 70 electron volts. The cycle time is adjusted to measure five or more spectra during the elution of each GC peak. A typical GC temperature program is described below, but is subject to change at the discretion of the analyst:

Initial Temperature:	35°C for 2 minutes.
Temperature Program:	35°C to 320°C at 14°/min.
Final Temperature:	320°C for 5.6 min or until Benzo (g,h,i) perylene has eluted
Injector Temperature:	250°C
Transfer Line Temperature:	300°C
Injector:	Grob-like, splitless
Injection volume:	2 µL
Carrier Gas:	Helium

11.3 Instrument control and acquisition parameters are defined on the ChemStation software for each instrument. Arrange the samples in the auto-sampler. Inject 2-uL of samples using the same instrument operating conditions that were used for initial calibration. Acquire the data and evaluate the results to confirm qualitative identification and quantification.

11.4 The data system tentatively identifies target analytes by comparing the retention time of the peaks to a window set around the daily calibration standard, and searches in that area for the primary and up to two secondary ions characteristic of the target analyte. All tentative identifications made by the computer are reviewed and either accepted or rejected by the analyst and/or data reviewer using the following criteria:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the user-created database. In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance if a tentative identification is to be made, and should be evaluated even if they are below 10% relative abundance.
- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

11.5 Identification is hindered when components are not chromatographically resolved from interfering analyte peaks or non-target constituents (background). When chromatographic peaks obviously indicate contribution from more than one component (broadened peak with shoulder(s) or a valley between two or more maxima), examine the EICPs to select the appropriate analyte spectra over the entire peak and use selective background subtraction in order to positively identify target analytes and account for extraneous ions. For coeluting compounds, the identification criteria will be met, but the analyte spectrum will contain extraneous ions contributed by the coeluting compound.

11.6 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs.

Complex environmental matrices, baseline upsets, coelution and peak shape variation can complicate automatic data system integration causing inaccurate and/or missed identification that should be corrected with manual integration. To assure accurate qualitative identification, optimize the data system integration parameters to ensure consistency in integration between standards and sample and evaluate each chromatogram to verify the identification for each target analyte. If necessary, perform manual integration to correct for data processing or integration error.

11.2.2 Tentatively Identified Compounds (TICs)

TICs may be reported upon client request. In general, TICs whose peak areas are > 10% of the nearest internal standard may be reported.

Perform the library search, and visually compare the sample spectra with the nearest library search and assign a tentative identification. The library search should not include peaks that are < 10% of the nearest internal standard, target analytes, or peaks that elute earlier than 30 seconds before the first target analyte.

The following criteria are used in qualitatively identifying these compounds:

- Relative intensities of ions greater than 10% of the most abundant ion in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

The laboratory may determine that a more general identification can be made, such as “unknown aliphatic compound, unknown aromatic compound, unknown cycloalkane, etc.”. TIC concentrations are calculated as outlined in Appendix A using an RF of 1.00. All TICs concentrations are reported with a “J” qualifier to indicate that the quantification is estimated. All cases of tentative identification are flagged with an “N” to indicate that there is presumptive evidence of a compound.

11.7 Quantification of Target Analytes

After a compound has been identified, the data system quantifies the concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP using the equations given in Appendix A. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are required, include this information in the non-conformance report and project narrative.

11.8 If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP LP-LB-0006 *Manual Integration.*

11.9 After analysis is complete, evaluate the results against the performance criteria given in Section 10 and Table 3, Section 18 and perform corrective action as necessary.

11.10 Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should ideally result in a determination within the upper half of the calibration curve.

12.0 CALCULATIONS

See Appendix A.

13.0 DATA REVIEW, CORRECTIVE ACTION & REPORTING

13.1 Review the samples, standards and QC samples against the acceptance criteria in Table 3. If the results do not fall within the established limits, perform the recommended corrective action. If corrective action is unsuccessful, document the situation with a nonconformance report and/or qualify the data using an appropriate data qualifier (see Section 13 for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

13.2 In the absence of project specific requirements, use the in-house statistically derived control limits specified in Table 5A & B. Table 5A also contains specific control limits that are required to be used for DoD projects.

Based on the number of analytes in the LCS (70-91), it is statistically likely that at least four analytes will marginally exceed the control limits; therefore, 4 marginal exceedances are allowed. A marginal exceedence (ME) is defined as being outside of the control limit of $\pm$ 3 SD, but within $\pm$ 4 SD. In order to easily calculate these limits, the following equation may be used:

$$\text{ME (Lower Limit)} = \text{Lower Limit} - \left(\frac{\text{Upper Limit} - \text{Lower Limit}}{6}\right)$$

$$\text{ME (Upper Limit)} = \text{Upper Limit} + \frac{(\text{Upper Limit} - \text{Lower Limit})}{6}$$

13.3 The following analytes have been identified as poorly performing analytes based on statistical data by both the DoD and the laboratory. Decisions regarding batch acceptability are not based on those analytes in the specific matrix identified. These are footnoted in Table 4.

Analyte	Based on:
Aqueous Matrix	
4-nitrophenol	DoD and Laboratory Data
Benzoic acid	DoD and Laboratory Data
Phenol	DoD and Laboratory Data
Phenol-d5	DoD and Laboratory Data
Benzidine	Laboratory Data[1]
2-Chlorobenzoic acid	Laboratory Data[1]
Hexachlorocyclopentadiene	Laboratory Data[1]
Pyridine	Laboratory Data[1]
Solids Matrix	
3,3'-Dichlorobenzidine	DoD and Laboratory Data
4-Chloroaniline	DoD and Laboratory Data
Benzoic acid	DoD and Laboratory Data
Aniline	Laboratory Data[1]
Benzidine	Laboratory Data[1]
2-Chlorobenzaldehyde	Laboratory Data[1]
2-Chlorobenzoic acid	Laboratory Data[1]
3-Chlorobenzaldehyde	Laboratory Data[1]
4-Chlorobenzaldehyde	Laboratory Data[1]
Pyridine	Laboratory Data[1]

[1] DoD data not available

13.4 Data Reporting

The laboratory's RL for each target analyte is provided in Table 1A & 1B. Report the data to the RL adjusted for sample matrix, percent moisture, and sample dilution/concentration. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results that have concentrations for a target analytes less than the RL with a "U" qualifier. Unless otherwise specified, report the results for solid matrices on a dry weight basis.

Some projects may require reporting positively identified target analytes less than the RL. In this case, the analyte can be qualitatively detected but not accurately quantified. Flag all results less than the RL with a "J" data qualifier.

Some projects may require RLs that are less than the laboratory's routine RL. Sample results may be reported to the project RL if the project RL is greater than the Quantification Limit (QL) and above the MDL. In this context, the QL is defined as the concentration of the low calibration standard. If the project RL is less than the QL, all values less than the QL must be reported as estimated and qualified with a "J".

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-LB-009 *Determination of Method Detection Limits.*

13.5 Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample
D = Compound is identified in an analysis at a secondary dilution factor
E = Compound quantification is above the instrument's calibration range for this analysis
J = Indicates an estimated quantification value
U = Compound was analyzed for but not detected
X = The reported compound is a suspected laboratory contaminant
Y = an additional qualifier which will be defined at the time of use by the data reviewer
Z = The reported result is based on the combined responses from coeluting compounds
* = Data outside of control limits.

13.6 Data Management and Records: All electronic and hardcopy data is managed, retained, and archived as specified in laboratory SOP LP-QA-0014 *Laboratory Records*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.4 The laboratory statistically derived control limits used to evaluate accuracy, precision and surrogate recoveries are provided in Table 2. The control limits for accuracy are based on compiled data and are set at 3 standard deviations around the mean using the procedures described in laboratory SOP LP-QA-012 *Control Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 The following waste streams are produced when this method is carried out:

- Vials containing sample extracts

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with

Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (Method 8270C), Revision 3, December 1996, USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

17.0 SOP REVISION HISTORY

The following changes were made in this revision:

Section 5:	Updated Safety section.
Section 7:	7.1 - Removed solvents not used in analytical method.
Section 10:	10.3 - Added additional quantification options. 10.4 - 10.6 Added detail about repeating CCV. 10.7 Added Troubleshooting.
Section 12:	Moved calculations to Appendix A. Moved data reporting to Section 13.
Section 13:	13.2 - Added detail regarding the use of DoD LCS and Surrogate Limits. 13.3 – Added list of poorly performing analytes. 13.4 - Added SOP reference for Data Management & Records. 13.5 – Added data flags.
Section 14:	Completely revised section.
Section 17:	New Section added.
Table 1A &B:	Added Footnotes. Eliminated Standard criteria. Added internal standard assignment.
Table 2:	Revised material list to solvents used in analysis only.
Table 3:	Changed DDT Breakdown Criteria from < 20% to $\leq$20%. Added phthalates as common laboratory contaminant.
Table 4:	Combined Table 4, 5, and 6, updated control limits, added Low Level Water and Soil Control Limits, added footnotes.
Table 5:	Eliminated original Table 5 (IS assignment and incorporated into Table 1.)
Table 5A&B:	Added footnotes, updated control limits, identified poorly performing analytes.
Appendix A:	New Appendix added containing all calculations.

18.0 TABLES, DIAGRAMS, & FLOWCHARTS

Table 1A:	Target Analyte List, RLs, Characteristic Ions, and IS Assignments
Table 1B:	A9 Extended Target Analyte List, RLs, Characteristic Ions, and IS Assignments
Table 2:	Primary Materials Used
Table 3:	QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action
Table 4:	DFTPP Key Ions and Abundance Criteria
Table 5A:	Control Limits as Accuracy (%R) and Precision (RPD)
Table 5B:	Control Limits as Accuracy (%R) and Precision (RPD) for Extended List

Table 1A: Target Analyte List, RLs, Characteristic Ions, and IS Assignments

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions			Internal Standard Assignment
		Water (ug/L)	Low Soil (ug/Kg)	Med Soil (ug/Kg)	Primary Ion	Secondary Ions		
Pyridine	110-86-1	10	330	10000	79	52	NA	1
N-Nitrosodimethylamine	62-75-9	10	330	10000	74	42	NA	1
Benzaldehyde	100-52-7	25	830	25000	77	105	106	1
Aniline	62-53-3	25	830	25000	93	66	65	1
bis(2-Chloroethyl)Ether	111-44-4	10	330	10000	93	95	NA	1
Phenol (CCC)	108-95-2	10	330	10000	94	65	66	1
2-Chlorophenol	95-57-8	10	330	10000	128	64	130	1
1,3-Dichlorobenzene	541-73-1	10	330	10000	146	148	111	1
1,4-Dichlorobenzene (CCC)	106-46-7	10	330	10000	146	148	111	1
1,2-Dichlorobenzene	95-50-1	10	330	10000	146	148	111	1
Benzyl Alcohol	100-51-6	10	330	10000	108	79	77	1
2,2'-oxybis(1-Chloropropane)	108-60-1	10	330	10000	45	121	NA	1
Acetophenone	98-86-2	10	330	10000	105	77	51	1
2-Methylphenol	95-48-7	10	330	10000	107	108	79	1
Hexachloroethane	67-72-1	10	330	10000	117	201	199	1
N-Nitroso-di-n-propylamine (SPCC)	621-64-7	10	330	10000	70	42	130	1
4-Methylphenol	106-44-5	10	330	10000	107	108	79	1
Nitrobenzene	98-95-3	10	330	10000	77	123	65	2
Isophorone	78-59-1	10	330	10000	82	95	138	2
2-Nitrophenol (CCC)	88-75-5	10	330	10000	139	109	65	2
2,4-Dimethylphenol	105-67-9	10	330	10000	122	107	121	2
bis(2-Chloroethoxy)methane	111-91-1	10	330	10000	93	95	123	2
2,4-Dichlorophenol	120-83-2	10	330	10000	162	164	98	2
Benzoic Acid	65-85-0	35	830	25000	105	122	77	2
1,2,4-Trichlorobenzene	120-82-1	10	330	10000	180	182	145	2
Naphthalene	91-20-3	10	330	10000	128	129	NA	2
4-Chloroaniline	106-47-8	10	330**	10000	127	129	NA	2
Hexachlorobutadiene (CCC)	87-68-3	10	330	10000	225	223	227	2
Caprolactum	105-60-2	10	330	10000	113	55	56	2
2-Methylnaphthalene	91-57-6	10	330	10000	115	141	142	2
4-Chloro-3-methylphenol	59-50-7	10	330	10000	107	144	NA	2
Hexachlorocyclopentadiene (SPCC)	77-47-4	10	330**	10000	237	235	272	3
2,4,6-Trichlorophenol	88-06-2	10	330	10000	196	198	200	3
2,4,5-Trichlorophenol	95-95-4	25	830	25000	196	198	97	3

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions			Internal Standard Assignment
		Water (ug/L)	Low Soil (ug/Kg)	Med Soil (ug/Kg)	Primary Ion	Secondary Ions		
2-Chloronaphthalene	91-58-7	10	330	10000	162	127	164	3
1,1'-Biphenyl	92-52-4	10	330	10000	154	153	152	3
2-Nitroaniline	88-74-4	25	830	25000	65	92	138	3
Acenaphthylene	208-96-8	10	330	10000	152	153	NA	3
Dimethylphthalate	131-11-3	10	330	10000	163	194	NA	3
2,6-Dinitrotoluene	606-20-2	10	330	10000	165	89	NA	3
Acenaphthene	83-32-9	10	330	10000	154	153	152	3
3-Nitroaniline	99-09-2	25	830	25000	138	65	92	3
2,4-Dinitrophenol (SPCC)	51-28-5	25	830	25000	187	107	NA	3
Dibenzofuran	132-64-9	10	330	10000	168	139	NA	3
2,4-Dinitrotoluene	121-14-2	10	330	10000	165	89	NA	3
4-Nitrophenol (SPCC)	100-02-7	25	830	25000	109	81	65	3
Fluorene	86-73-7	10	330	10000	166	165	NA	3
Diethylphthalate	84-66-2	10	330	10000	149	177	150	3
4-Chlorophenyl-phenylether	7005-72-3	10	330	10000	204	206	141	3
4-Nitroaniline	100-01-6	25	830	25000	138	92	108	3
4,6-Dinitro-2-methylphenol	534-52-1	25	830	25000	198	121	106	4
Azobenzene	103-33-3	10	830	25000	182	77	NA	4
N-nitrosodiphenylamine[2] (CCC)	86-30-6	10	330	10000	169	168	167	4
4-Bromophenyl-phenylether	101-55-3	10	330	10000	248	250	141	4
Hexachlorobenzene	118-74-1	10	330	10000	284	142	249	4
Atrazine	1912-24-9	10	330	10000	200	173	215	4
Pentachlorophenol (CCC)	87-86-5	25	830	25000	266	268	204	4
Phenanthrene	85-01-8	10	330	10000	178	179	176	4
Anthracene	120-12-7	10	330	10000	178	176	179	4
Carbazole	86-74-8	10	330	10000	167	139	NA	4
Di-n-butylphthalate	84-74-2	10	330	10000	149	150	104	4
Fluoranthene (CCC)	206-44-0	10	330	10000	202	203	101	4
Pyrene	129-00-0	10	330	10000	202	203	101	5
Benzidine	92-87-5	25	830	25000	184	92	185	5
Butylbenzylphthalate	85-68-7	10	330	10000	149	91	206	5
Benzo(a)anthracene	56-55-3	10	330	10000	228	229	226	5
Chrysene	218-01-9	10	330	10000	228	226	229	5
3,3'-Dichlorobenzidine	91-94-1	10	330	10000	252	254	126	5
bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10000	149	167	NA	5
Di-n-octylphthalate (CCC)	117-84-0	10	330	10000	149	NA	NA	6

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions			Internal Standard Assignment
		Water (ug/L)	Low Soil (ug/Kg)	Med Soil (ug/Kg)	Primary Ion	Secondary Ions		
Benzo(b)fluoranthene	205-99-2	10	330	10000	252	253	125	6
Benzo(k)fluoranthene	207-08-9	10	330	10000	252	253	125	6
Benzo(a)pyrene	50-32-8	10	330	10000	252	253	125	6
Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10000	276	138	277	6
Dibenz(a,h)anthracene	53-70-3	10	330	10000	278	139	279	6
Benzo(g,h,i)perylene	191-24-2	10	330	10000	276	138	277	6
Surrogate Compounds:								
2-Fluorophenol	367-12-4	NA	NA	NA	112	64	NA	1
Phenol-d5	4165-62-2	NA	NA	NA	99	71	42	1
2-Chlorophenol-d4	93951-73-6	NA	NA	NA	132	68	134	1
1,2-Dichlorobenzene-d4	2199-69-1	NA	NA	NA	152	150	115	1
Nitrobenzene-d5	4165-60-0	NA	NA	NA	82	54	128	2
2-Fluorobiphenyl	321-60-8	NA	NA	NA	172	171	NA	3
2,4,6-Tribromophenol	118-79-6	NA	NA	NA	330	332	141	4
Terphenyl-d14	98904-43-9	NA	NA	NA	244	122	212	5
Internal Standards:								
1,4-Dichlorobenzene-d4 (IS1)	3855-82-1	NA	NA	NA	152	115	150	NA
Naphthalene-d8 (IS2)	1146-65-2	NA	NA	NA	136	68	NA	NA
Acenaphthene-d10 (IS3)	15067-26-2	NA	NA	NA	164	162	160	NA
Phenanthrene-d10 (IS4)	1517-22-2	NA	NA	NA	188	94	80	NA
Chrysene-d12 (IS5)	1719-03-5	NA	NA	NA	240	120	236	NA
Perylene-d12 (IS6)	1520-96-3	NA	NA	NA	264	260	265	NA

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.

[2] Analyzed as Diphenylamine due to breakdown in the analytical portion of the procedure.

CCC: Calibration Check Compound

SPCC: System Performance Check Compound

Table 1B: A9 Extended Target Analyte List, RLs, Characteristic Ions, and IS Assignments

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions		
		Water (ug/L)	Low Soil (ug/Kg)	Soil (ug/Kg)	Primary Ion	Secondary Ions	
Ethyl methacrylate	97-63-2	10	330	10000	69	99	41
2-Picoline	109-06-8	10	330	10000	93	66	NA
N-Nitrosomethyl-ethylamine	10595-95-6	10	330	10000	88	42	43
Methyl methanesulfonate	66-27-3	10	330	10000	80	79	65
N-Nitrosodiethylamine	55-18-5	10	330	10000	102	57	44
Ethyl methanesulfonate	62-50-0	10	330	10000	79	109	97
Pentachloroethane	76-01-7	10	330	10000	117	167	83
N-Nitrosopyrrolidine	930-55-2	10	330	10000	100	68	42
o-Toluidine	108-44-1	10	330	10000	106	107	NA
N-Nitrosomorpholine	59-89-2	10	330	10000	56	86	116
N-Nitrosopiperidine	100-75-4	10	330	10000	114	42	55
a,a-Dimethylphenethylamine	122-09-8	10	330	10000	58	91	NA
O,O,O-Triethylphosphorothioate	126-68-1	10	330	10000	198	121	97
p-Phenylenediamine	106-50-3	10	330	10000	108	80	53
Isosafrole	120-58-1	10	330	10000	162	104	131
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330	10000	216	214	218
Safrole	94-59-7	10	330	10000	104	103	131
1,4-Naphthoquinone	130-15-4	10	330	10000	158	102	76
m-Dinitrobenzene	99-65-0	10	330	10000	168	50	76
Pentachlorobenzene	608-93-5	10	330	10000	250	248	215
1-Naphthylamine	134-32-7	10	330	10000	143	116	115
2-Naphthylamine	91-59-8	10	330	10000	143	116	115
2,3,4,6-Tetrachlorophenol	58-90-2	10	330	10000	232	230	131
5-Nitro-o-toluidine	99-55-8	10	330	10000	152	106	79
Thionazin	297-97-2	10	330	10000	97	107	143
Sulfotepp	3689-24-5	10	330	10000	322	202	266
Diallate	2303-16-4	10	330	10000	86	234	43
Phorate	298-02-2	10	330	10000	121	260	NA
sym-Trinitrobenzene	99-35-4	10	330	10000	213	74	91
Phenacetin	62-44-2	10	330	10000	108	179	137
Dimethoate	60-51-5	10	330	10000	87	125	93
4-Aminobiphenyl	92-67-1	10	330	10000	169	168	115
Pentachloronitrobenzene	82-68-8	10	330	10000	237	214	142
Pronamide	23950-58-6	10	330	10000	173	175	145
Disulfoton	298-04-4	10	330	10000	97	142	NA

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions		
		Water (ug/L)	Low Soil (ug/Kg)	Soil (ug/Kg)	Primary Ion	Secondary Ions	
Dinoseb	88-85-7	10	330	10000	211	163	147
Methyl parathion	298-00-0	10	330	10000	109	125	263
4-Nitroquinoline-1-oxide	56-57-5	10	330	10000	101	128	174
Ethyl parathion	56-38-2	10	330	10000	109	97	291
Methapyrilene	91-80-5	10	330	10000	58	97	71
Isodrin	465-73-6	10	330	10000	193	147	66
Aramite	140-57-8	10	330	10000	185	191	319
p-(Dimethylamino)Azobenzene	60-11-7	10	330	10000	120	225	77
Chlorobenzilate	510-15-6	10	330	10000	251	139	111
Kepone	143-50-0	10	830	10000	272	237	143
3,3'-Dimethylbenzidine	119-93-7	10	330	10000	212	213	106
2-Acetylaminofluorene	53-96-3	10	330	10000	181	223	152
7,12-Dimethylbenz(a)anthracene	57-97-6	10	330	10000	256	241	128
3-Methylcholanthrene	56-49-5	10	330	10000	268	253	126
Ethyl methacrylate	97-63-2	10	330	10000	69	99	41
2-Picoline	109-06-8	10	330	10000	93	66	NA
N-Nitrosomethyl-ethylamine	10595-95-6	10	330	10000	88	42	43
Methyl methanesulfonate	66-27-3	10	330	10000	80	79	65
N-Nitrosodiethylamine	55-18-5	10	330	10000	102	57	44
Ethyl methanesulfonate	62-50-0	10	330	10000	79	109	97

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Minimum Frequency	Acceptance Criteria	Recommended Corrective Action[1]
DFTPP	Before initial and continuing calibration, every 12 hours	See Table 4	Reshoot, retune mass spectrometer
DDT Breakdown Check	Daily prior to sample analysis (DoD Only)	Degradation $\leq$20%	Correct problem and repeat check; no samples may be analyzed until acceptance criterion is met.
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CCCs: %RSD $\leq$ 30% SPCCs: mean RF $\geq$ 0.050. Linear Regression: r $\geq$ 0.99 (0.995 for DoD) Quadratic: $r^2 \geq 0.99$	Correct problem and repeat calibration.
ICV	After each initial calibration	%Difference ± 25%	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Beginning of each 12-hour window, as established by a compliant DFTPP.	SPCCs: mean RF $\geq$ 0.050. CCCs: %D ≤ 20%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs at different concentrations pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	< RL DoD: $\leq$ ½ RL for all analytes except < RL for phthalates for any sample $\geq$ RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	Evaluated against control limits in Table 5, 4 Marginal Exceedances allowed.	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD	One per extraction batch of 20 or less samples.	Evaluated against control limits in Table 5	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate Standard	All field and QC samples	Evaluated against control limits in Table 5	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.
Internal Standard	All field and QC samples	Area between 50-200% of area of daily calibration internal standard area	Same as above.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that data quality is known and documented. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Table 4: DFTPP Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
69	Present
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40.0 of mass 198
443	17-23% of mass 442

Table 5A: Control Limits[1] as Accuracy (%R) and Precision[2] (RPD)

Analyte	Water				Solid			
	Lab Limits		DoD Limits		Lab Limits		DoD Limits	
	%R	RPD	%R	RPD	%R	RPD	%R	RPD
Pyridine	10-105(5)	50	10-105(3,5)	50	15-105(5)	50	15-105(3,5)	50
N-Nitrosodimethylamine	45-110	30	25-110	30	55-125	30	20-115	30
Benzaldehyde	60-140(6)	30	60-140(6)	30	60-140(6)	30	60-140(6)	30
Aniline	10-110	30	10-110(3)	30	10-105(5)	50	10-105(3,5)	50
bis(2-Chloroethyl)Ether	70-140	30	35-110	30	60-125	30	40-105	30
Phenol	25-90(4)	30	25-90(3,4)	30	**60-140**	30	40-100	30
2-Chlorophenol	**65-140**	30	65-135	30	60-125	30	45-105	30
1,3-Dichlorobenzene	50-125	30	30-100	30	**50-115**	30	40-100	30
1,4-Dichlorobenzene	55-125	30	30-100	30	55-120	30	35-105	30
1,2-Dichlorobenzene	55-130	30	35-100	30	55-120	30	45-95	30
Benzyl Alcohol	**45-150**	30	30-110	30	50-155	30	20-125	30
2,2'-oxybis(1-Chloropropane)	60-150	30	25-130	30	60-130	30	20-115	30
Acetophenone	60-140(6)	30	60-140(6)	30	60-140(6)	30	60-140(6)	30
2-Methylphenol	**55-130**	30	40-110	30	**50-135**	30	40-105	30
Hexachloroethane	**50-140**	30	30-95	30	55-120	30	35-110	30
N-Nitroso-di-n-propylamine	60-130	30	35-130	30	**45-125**	30	40-115	30
4-Methylphenol	**35-130**	30	30-110	30	**35-140**	30	40-105	30
Nitrobenzene	**60-135**	30	45-110	30	55-120	30	40-115	30
Isophorone	**45-135**	30	50-110	30	50-115	30	45-110	30
2-Nitrophenol	70-145	30	40-115	30	**55-135**	30	40-110	30
2,4-Dimethylphenol	**30-165**	30	30-110	30	**25-150**	30	30-105	30
bis(2-Chloroethoxy)methane	**45-160**	30	45-105	30	**55-125**	30	45-110	30
2,4-Dichlorophenol	55-150	30	50-105	30	**55-130**	30	45-110	30
Benzoic Acid	10-70(4)	50	10-70(3,4)	50	25-145(3,4)	50	25-145(3,4)	50
1,2,4-Trichlorobenzene	60-130	30	35-105	30	55-120	30	45-110	30
Naphthalene	**65-135**	30	40-100	30	55-120	30	40-105	30
4-Chloroaniline	**10-95**(5)	30	15-110	30	10-90(3,4)	50	10-90(3,4)	50
Hexachlorobutadiene	**35-140**	30	25-105	30	**50-130**	30	40-115	30
Caprolactum	60-140(6)	30	60-140(6)	30	60-140(6)	30	60-140(6)	30
2-Methylnaphthalene	50-145	30	45-105	30	**45-150**	30	45-105	30
4-Chloro-3-Methylphenol	**50-155**	30	45-110	30	**60-140**	30	45-115	30
Hexachlorocyclopentadiene	10-155(5)	50	10-155(3,5)	50	30-105(3)	40	30-105(3)	40
2,4,6-Trichlorophenol	**55-155**	30	45-110	30	**60-135**	30	45-110	30
2,4,5-Trichlorophenol	**60-140**	30	60-120	30	**60-130**	30	50-110	30
2-Chloronaphthalene	**50-125**	30	50-105	30	**45-120**	30	45-105	30
1,1'-Biphenyl	60-140(6)	30	60-140(6)	30	60-140(6)	30	60-140(6)	30
2-Nitroaniline	**65-140**	30	65-130	30	50-125	30	45-120	30
Acenaphthylene	**60-125**	30	50-105	30	**50-115**	30	45-105	30

Analyte	Water				Solid			
	Lab Limits		DoD Limits		Lab Limits		DoD Limits	
	%R	RPD	%R	RPD	%R	RPD	%R	RPD
Dimethylphthalate	65-140	30	25-125	30	55-120	30	50-110	30
2,6-Dinitrotoluene	**70-135**	30	50-115	30	**55-130**	30	50-110	30
Acenaphthene	**60-135**	30	45-110	30	55-120	30	45-110	30
3-Nitroaniline	30-95	30	20-125	30	**20-85**	30	25-110	30
2,4-Dinitrophenol	35-150	30	15-140	30	**25-160**	30	15-130	30
Dibenzofuran	**65-140**	30	55-105	30	**55-120**	30	50-105	30
2,4-Dinitrotoluene	60-130	30	50-120	30	**50-120**	30	50-115	30
4-Nitrophenol	10-110(4)	30	10-110(3,4)	30	30-155	30	15-140	30
Fluorene	**65-135**	30	50-110	30	**50-125**	30	50-110	30
Diethylphthalate	**55-140**	30	40-120	30	**50-125**	30	50-115	30
4-Chlorophenyl-phenylether	**60-140**	30	50-110	30	**50-120**	30	45-110	30
4-Nitroaniline	50-135	30	35-120	30	**25-115**	30	35-115	30
4,6-Dinitro-2-methylphenol	**55-180**	30	40-130	30	55-160	30	30-135	30
N-nitrosodiphenylamine	65-125	30	50-110	30	**45-130**	30	50-115	30
Azobenzene	65-135(3)	35	65-135(3)	35	60-140	30	60-140(6)	30
4-Bromophenyl-phenylether	**55-150**	30	50-115	30	**55-130**	30	45-115	30
Hexachlorobenzene	**55-150**	30	50-110	30	55-125	30	45-120	30
Atrazine	60-140(6)	30	60-140(6)	30	60-140(6)	30	60-140(6)	30
Pentachlorophenol	**50-165**	30	40-115	30	50-140	30	25-120	30
Phenanthrene	**70-135**	30	50-115	30	**60-125**	30	50-110	30
Anthracene	**70-135**	30	55-110	30	**60-125**	30	55-105	30
Carbazole	**60-140**	30	50-115	30	55-125	30	45-115	30
Di-n-butylphthalate	**60-135**	30	55-115	30	**50-120**	30	55-110	30
Fluoranthene	50-140	30	55-115	30	**50-120**	30	55-115	30
Pyrene	**70-160**	30	50-130	30	**35-175**	30	45-125	30
Benzidine	10-235(5)	50	10-235(3,5)	50	10-120(5)	50	10-120(3,5)	50
Butylbenzylphthalate	75-140	30	45-115	30	65-145	30	50-125	30
Benzo(a)anthracene	**70-135**	30	55-110	30	**55-130**	30	50-110	30
Chrysene	65-130	30	45-120	30	**60-125**	30	55-110	30
3,3'-Dichlorobenzidine	**10-140**	30	20-110	30	10-120(4)	50	10-120(3,4)	50
bis(2-Ethylhexyl)phthalate	80-145	30	40-125	30	**55-140**	30	45-125	30
Di-n-octylphthalate	70-135	30	35-135	30	60-135	30	40-130	30
Benzo(b)fluoranthene	**40-150**	30	45-120	30	**45-130**	30	45-115	30
Benzo(k)fluoranthene	60-140	30	45-125	30	60-125	30	45-125	30
Benzo(a)pyrene	**65-130**	30	55-110	30	55-120	30	50-110	30
Indeno(1,2,3-cd)pyrene	**45-150**	30	45-125	30	55-135	30	40-120	30
Dibenz(a,h)anthracene	**45-150**	30	40-125	30	35-145	30	40-125	30
Benzo(g,h,i)perylene	**50-140**	30	40-125	30	**45-135**	30	40-125	30
Surrogates								
2-Fluorophenol	30-95	NA	20-110	NA	45-110	NA	35-105	NA

Analyte	Water				Solid			
	Lab Limits		DoD Limits		Lab Limits		DoD Limits	
	%R	RPD	%R	RPD	%R	RPD	%R	RPD
Phenol-d5	10-75[4]	NA	10-75 [3,4]	NA	**50-120**	NA	40-100	NA
2-Chlorophenol-d4	55-120	NA	55-120[3]	NA	50-115	NA	50-115[3]	NA
1,2-Dichlorobenzene-d4	55-120	NA	55-120[3]	NA	50-115	NA	50-115[3]	NA
Nitrobenzene-d5	55-125	NA	40-110	NA	50-115	NA	35-100	NA
2-Fluorobiphenyl	30-95	NA	50-110	NA	**45-110**	NA	45-105	NA
2,4,6-Tribromophenol	50-135	NA	40-125	NA	50-120	NA	35-125	NA
Terphenyl-d14	**55-145**	NA	50-135	NA	**40-145**	NA	30-125	NA

(1) The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.
(2) RPD for MS/MSD only.
(3) DoD Limit not available so limit is statistically derived laboratory limit.
(4) Identified as poorly performing analyte by DoD. Decisions regarding batch acceptability are not based on analyte in the specific matrix indicated.
(5) Identified as poorly performing analyte by laboratory. Decisions regarding batch acceptability are not based on analyte in the specific matrix indicated.
(6) Default limit.
Note: Where in house limits are outside of DoD limits, this is indicated in bold.

Table 5B: Control Limits[1,2,4] as Accuracy (%R) and Precision[3] (RPD) for Extended List

Analyte	CAS #	Water		Soil	
		%R	RPD	%R	RPD
Ethyl methacrylate	97-63-2	60-140	30	60-140	30
2-Picoline	109-06-8	60-140	30	60-140	30
N-Nitrosomethyl-ethylamine	10595-95-6	60-140	30	60-140	30
Methyl methanesulfonate	66-27-3	60-140	30	60-140	30
N-Nitrosodiethylamine	55-18-5	60-140	30	60-140	30
Ethyl methanesulfonate	62-50-0	60-140	30	60-140	30
Pentachloroethane	76-01-7	60-140	30	60-140	30
N-Nitrosopyrrolidine	930-55-2	60-140	30	60-140	30
o-Toluidine	108-44-1	60-140	30	60-140	30
N-Nitrosomorpholine	59-89-2	60-140	30	60-140	30
N-Nitrosopiperidine	100-75-4	60-140	30	60-140	30
a,a-Dimethylphenethylamine	122-09-8	60-140	30	60-140	30
O,O,O-Triethylphosphorothioate	126-68-1	60-140	30	60-140	30
p-Phenylenediamine	106-50-3	60-140	30	60-140	30
Isosafrole	120-58-1	60-140	30	60-140	30
1,2,4,5-Tetrachlorobenzene	95-94-3	60-140	30	60-140	30
Safrole	94-59-7	60-140	30	60-140	30
1,4-Naphthoquinone	130-15-4	60-140	30	60-140	30
m-Dinitrobenzene	99-65-0	60-140	30	60-140	30
Pentachlorobenzene	608-93-5	60-140	30	60-140	30
1-Naphthylamine	134-32-7	60-140	30	60-140	30
2-Naphthylamine	91-59-8	60-140	30	60-140	30
2,3,4,6-Tetrachlorophenol	58-90-2	60-140	30	60-140	30
5-Nitro-o-toluidine	99-55-8	60-140	30	60-140	30
Thionazin	297-97-2	60-140	30	60-140	30
Sulfotepp	3689-24-5	60-140	30	60-140	30
Diallate	2303-16-4	60-140	30	60-140	30
Phorate	298-02-2	60-140	30	60-140	30
sym-Trinitrobenzene	99-35-4	60-140	30	60-140	30
Phenacetin	62-44-2	60-140	30	60-140	30
Dimethoate	60-51-5	60-140	30	60-140	30
4-Aminobiphenyl	92-67-1	60-140	30	60-140	30
Pentachloronitrobenzene	82-68-8	60-140	30	60-140	30
Pronamide	23950-58-6	60-140	30	60-140	30
Disulfoton	298-04-4	60-140	30	60-140	30
Dinoseb	88-85-7	60-140	30	60-140	30
Methyl parathion	298-00-0	60-140	30	60-140	30
4-Nitroquinoline-1-oxide	56-57-5	60-140	30	60-140	30
Ethyl parathion	56-38-2	60-140	30	60-140	30

Analyte	CAS #	Water		Soil	
		%R	RPD	%R	RPD
Methapyrilene	91-80-5	60-140	30	60-140	30
Isodrin	465-73-6	60-140	30	60-140	30
Aramite	140-57-8	60-140	30	60-140	30
p-(Dimethylamino)Azobenzene	60-11-7	60-140	30	60-140	30
Chlorobenzilate	510-15-6	60-140	30	60-140	30
Kepone	143-50-0	60-140	30	60-140	30
3,3'-Dimethylbenzidine	119-93-7	60-140	30	60-140	30
2-Acetylaminofluorene	53-96-3	60-140	30	60-140	30
7,12-Dimethylbenz(a)anthracene	57-97-6	60-140	30	60-140	30
3-Methylcholanthrene	56-49-5	60-140	30	60-140	30

[1] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.
[2] Limits are default.
[3] RPD for MS/MSD only.

Appendix A: Equations

Response Factor (RFx) = $\dfrac{Area_x \text{ X } Concentration_{is}}{Area_{is} \text{ X } Concentration_x}$

Where: x=compound, is = Internal Standard

Relative Retention Time (RRT) = $\dfrac{\text{Retention Time}_x}{\text{Retention Time}_{is}}$

where: x=compound, is = Internal Standard

Mean Response Factor ($\overline{RF}$) = $\dfrac{\sum_{i=1}^{n} RF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Response Factor (SD) = $\sqrt{\dfrac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Response = $\dfrac{SD}{\overline{\overline{RF}}} \times 100\%$

Percent Difference (%D) = $\dfrac{RF_v - \overline{RF}}{\overline{\overline{RF}}} \times 100\%$

where: RF_v = Response Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\dfrac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ X 100%

Percent Recovery (%R) = $\dfrac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration (for average RF quantification)

Aqueous Samples

$$C_x = \frac{A_x \times C_{IS} \times V_t}{A_{IS} \times MeanRF \times V_o \times V_i} \times DF$$

Where:

C_x	=	Concentration of compound (ug/L)
A_x	=	Area of quantitation ion
C_{IS}	=	Concentration of associated internal standard (ng)
V_t	=	Extract Volume (uL)
A_{IS}	=	Area of quantitation ion for associated internal standard.
Mean RF	=	Mean Response Factor from initial calibration, or 1 for a TIC or Alkane
V_o	=	Sample volume (mL)
V_I	=	Volume injected (uL)
DF	=	Dilution Factor.

Solid Samples

$$C_x = \frac{A_x \times C_{IS} \times V_t \times GPC \times 10^3 g/Kg}{A_{IS} \times MeanRF \times W_s \times S \times V_i \times 10^3 ng/ug} \times DF$$

Where:

C_x	=	Concentration of compound (ug/Kg)
A_x	=	Area of quantitation ion for compound.
A_{IS}	=	Area of quantitation ion for associated internal standard.
C_{IS}	=	Concentration of associated internal standard (ng)
V_t	=	Volume of final extract (uL)
GPC	=	GPC Factor
Mean RF	=	Mean Response Factor from initial calibration, or 1 for a TIC or Alkane
W_s	=	Weight of sample (g)
S	=	Percent Solids (as a decimal)
V_i	=	Volume injected (uL)
DF	=	Dilution Factor

Sample Concentration (for linear regression or quadratic equation quantification)

Aqueous Samples

$$C_x = \frac{Ce \times V_t}{V_o \times V_i} \times DF$$

Where:

C_x	=	Concentration of compound (ug/L)
Ce	=	ng analyte from the curve
V_t	=	Extract Volume (uL)
V_o	=	Sample volume (mL)
V_i	=	Volume injected (uL)
DF	=	Dilution Factor.

Solid Samples

$$C_x = \frac{Ce \times V_t \times GPC \times 10^3 g/Kg}{W_s \times S \times V_i \times 10^3 ng/ug} \times DF$$

Where:

C_x	=	Concentration of compound (ug/Kg)
Ce	=	ng analyte from the curve
GPC	=	GPC Factor
W_s	=	Weight of sample (g)
S	=	Percent Solids (as a decimal)
V_i	=	Volume injected (uL)
DF	=	Dilution Factor

Appendix B: Terms & Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed.

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Calibration Check Compounds (CCCs): Selective analytes from the compound list that are used to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Internal Standard: a known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery of the for each analyte.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a <u>range</u> where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

System Performance Check Compounds (SPCCs): Selective analytes from the compound list that are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.

STANDARD OPERATING PROCEDURE
ACID DIGESTION OF SOILS, SEDIMENTS & SLUDGE FOR TOTAL METALS
ICP-AES AND ICP-MS
SW-846 3050

Applicable Matrices: Sediment, Sludge, Soil, Tissue, Filters & Wipes

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APPROVAL SIGNATURES

Laboratory Director:	Michael F. Wheeler, Ph.D.	Date: 5/18/05
QA Manager:	Kirstin L. McCracken	Date: 5/18/05
Department Manager:	William S. Cicero	Date: 5/17/05

Proprietary Information Statement:

1.0 SCOPE AND APPLICATION

1.1. This SOP describes the laboratory procedure for the preparation of soil, sediment, tissue, air filter & wipe samples for the determination of total metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) or Inductively Coupled Atomic Plasma-Mass Spectrometry (ICP-MS). Samples prepared by this procedure may be analyzed for the list of metals given in Table 1, Section 17.0. Separate procedures are given for each analytical system because the extracts for ICP-AES and ICP-MS are not interchangeable.

2.0 SUMMARY OF METHOD

2.1. A representative 1-2 gram (wet weight) soil, sediment or tissue sample is digested with repeated additions of nitric acid and hydrogen peroxide. Whole or sub-samples of air filter or wipe samples may also be digested. For ICP-MS analysis, the resultant digestate is reduced in volume while heating and then diluted to a final volume of 100 mL. For ICP-AES analysis, hydrochloric acid (HCl) is added to the initial digestate, the sample is refluxed, filtered and the digestate is diluted to a final volume of 100 mL.

2.2. The procedure is based on Method 3050B Test methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition September 1986.

3.0 DEFINITIONS

3.1. Definitions are included in Appendix B.

4.0 INTERFERENCES

4.1. The metals digestion area should be free of dust, particulates, and materials from fume hood because these can contribute to contamination.

4.2. The data user should be cautioned that this digestion procedure may not be sufficiently vigorous to destroy some metal complexes and precipitation in the digestate may results in a low biased silver concentration.

5.0 SAFETY

5.1. Employees must trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Specific Safety Concerns & Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. Primary Materials Used

Table 2, Section 17.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1. Block Digester: Environmental Express, 25 Position Digester or equivalent. Capable of maintaining temperature of 90-95°C

6.2. Polyethylene Digestion Vessel (100 mL) ; purchased from Environmental Express

6.3. Volumetric Pipettes (Finpipette Adjustable Pipettes; Sizes 0.10-1.00 mL & 1.00-5.00 mL or equivalent.

6.4. Class A Volumetric Flasks; 50, 100, 500, and 1000 mL sizes

6.5. Top Loading Balance: capable of measurements to 0.1 g

6.6. Polyethyelene Ribbed Watch Glass

6.7. Whatman Filter Paper, No. 42 or equivalent

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water

30% Hydrogen Peroxide (H_2O_2), Standard grade; J.T. Baker or equivalent

30% Hydrogen Peroxide (H_2O_2), High Purity grade; J.T. Baker or equivalent (when analysis for Sn is required)

Nitric Acid (HNO_3); concentrated, reagent grade; J.T. Baker or equivalent

1:1 Nitric Acid: Fill container half full with Nanopure water and then add concentrated HNO_3 until up to full mark. Shake to mix reagent. Solution is now ready for use.

Hydrochloric Acid (HCl); concentrated, reagent grade; J.T. Baker or equivalent

7.2 Standards

The primary source standards are purchased from SPEX. The second source standards are purchased from Inorganic Ventures. Store the standards at room temperature and use until manufacturers assigned expiration date.

Prepare working standard solutions as needed, every 6 months or in accordance with the expiration date of the parent standard, whichever occurs first.

Matrix Spiking Solution: Prepare a mixed element solution by diluting known volumes of certified stock standard solutions in reagent water. The components, recipe and final concentration in digestate are provided in Appendix A.

Laboratory Control Sample (LCS) Solution: Prepare a mixed element solution by diluting known volumes of prepared intermediate standard solutions in reagent water. The components, recipe and final concentration in digestate are provided in Appendix A.

8.0 SAMPLE HANDLING, PRSERVATION, SHIPMENT & STORAGE

8.1 A minimum of 5 grams of sample should be collected in polyethylene or glass containers. Soil samples do not require chemical or thermal preservation.

8.2. The holding time is 180 days from date of collection.

8.3. Unless otherwise specified by client or regulatory program, after digestion and analysis, samples are held for a minimum of 30 days and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1. A method blank and lab control sample (LCS) must be performed with each digestion batch of 20 or fewer samples. For air filter or wipe samples, the method blank and LCS should contain clean filter or wipe material equivalent in size to that used for sample collection. A matrix spike (MS) and sample duplicate (DP) are prepared per batch of 20 or fewer samples, provided sufficient sample volume is available.

9.2. The LCS and MS must be fortified with spike solution prior to digestion. Whenever possible, the spike solution should include all elements of interest to the project or contract.

9.3. The criteria used to assess quality control samples are given in the analytical SOP for the determinative method.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Check the calibration of the balance each day of use prior to use with at least 3 Class S weights that bracket the range of use.

10.2 Check the accuracy of the mechanical pipettes each day of use prior to use.

11.0 PROCEDURE

11.1. ICP-AES Sample Preparation

Mix the sample thoroughly and weigh a 1-2 g portion of sample to the nearest 0.01 g and transfer the aliquot to a digestion vessel. For filter or wipe samples, use a portion of or the entire sample. Use 1-2 g of reagent water for the method blank.

To prepare the LCS, transfer 1.0 mL of the ICP-AES matrix spike solution and 1.0 mL of the Supplemental Solid LCS Solution into a digestion vessel. To prepare the matrix spike, weigh a 1-2 gram portion of sample to a polyethylene digestion vessel and add 1.0 mL of the ICP-AES matrix spike solution.

Add 10 mL of 1:1 HNO_3 to each digestion vessel, mix the slurry, and cover with a watch glass. Reflux the sample at 90-95°C for 10 minutes without boiling. Allow the sample to cool, add 5mL of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, repeat this step (5mLs concentrated HNO_3 followed by refluxing for 30 minutes) until fuming is no longer observed. Continue to heat the solution without boiling for 2 hours. Do not allow the solution to evaporate to dryness.

Cool the digestate and add 2 mL of reagent water and 3mL of 30% H_2O_2. Cover with a watch glass and return to the heat source to start the peroxide reaction. Heat until effervescence subsides, and cool the digestion vessel. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until general sample appearance is unchanged. Do not add more than a total of 10 mL 30% H_2O_2.

Note: If samples require analysis for Sn, high purity H_2O_2 must be used. Standard purity H_2O_2 has been found to contain quantifiable levels of Sn.

Cover the sample with a watch glass and continue to heat the acid-peroxide digestate until the apparent volume is reduced to ~5 mL. Do not allow the solution to evaporate to dryness.

Add 10 mL of concentrated HCl, replace watch glass, and reflux at 90-95°C for 15 minutes. Filter the digestate through a Whatman No. 42 filter paper, collect the filtrate in a 100 mL volumetric flask and adjust to volume with reagent water.

11.2 ICP-MS Sample Preparation.

Mix the sample thoroughly and weigh a 1-2 g portion of sample to the nearest 0.01 grams and transfer the aliquot to a digestion vessel. For filter or wipe samples, use a portion of or the entire sample. Use 1-2 g of reagent water for the method blank.

To prepare the LCS, transfer 1.0 mL of the ICP-MS matrix spike solution. To prepare the matrix spike, weigh a 1-2 gram portion of sample to a polyethylene digestion vessel and add 0.5 mL of the ICP-MS matrix spike solution.

Add 10 mL of 1:1 HNO_3 to each digestion vessel, mix the slurry, and cover with a watch glass. Reflux the sample at 90-95°C for 10 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, repeat this step (5 mL concentrated HNO_3 followed by refluxing for 30 minutes) until fuming is no longer observed. Continue to heat the solution without boiling for 2 hours. Do not allow the solution to evaporate to dryness.

Cool the digestate and add 2 mL of reagent water and 3 mL of 30% H_2O_2. Cover with a watch glass and return to the heat source to start the peroxide reaction. Heat until effervescence subsides, and cool the digestion vessel. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until general sample appearance is unchanged. Do not add more than a total of 10 mL 30% H_2O_2.

Cover the sample with a watch glass and continue to heat the acid-peroxide digestate until the apparent volume is reduced to ~5 mL. Do not allow the solution to evaporate to dryness.

Cool and dilute to 100 mL with reagent water.

If particulates are present in the digestate, filter through a Whatman No.41 filter paper and collect the filtrate, adjust to volume. In lieu of filtration, the digestates may be centrifuged or allowed to set overnight to settle suspended particulate matter.

12.0 CALCULATIONS

Not Applicable

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1. The digestion log is completed by the analyst(s) that performed the procedure and reviewed for completeness by the department supervisor or a secondary data reviewer. Problems encountered during the digestion process are documented on the digestion log or with a nonconformance report and the situation is described in the case narrative provided with the data package report.

14.0 METHOD PERFORMANCE

14.1. A demonstration of analyst capability (IDOC) is required prior to use of this SOP and any time there is a significant change in instrument type, personnel or test method. IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training.*

14.2. A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009, *Method Detection Limits & Instrument Detection Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1. Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Acidic Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

16.1. Method 3050B, Test methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition September 1986.

17.0 TABLES, DIAGRAMS & FLOWCHARTS

17.1. Table 1: Target Element List
17.2. Table 2: Primary Materials Used
17.3 Appendix A: Standard Tables
17.4 Appendix B: Terms and Definitions

Table 1: Target Element List

Metal	CAS #	ICP-MS isotopes
Aluminum	7429-90-5	27
Antimony	7440-36-0	123
Arsenic	7440-38-2	75
Barium	7440-39-3	135
Beryllium	7440-41-7	9
Boron[1]	7440-42-8	11
Cadmium	7440-43-9	111
Calcium	7440-70-2	44
Chromium	7440-47-3	52
Cobalt	7440-48-4	59
Copper	7440-50-8	65
Iron	7439-89-6	54
Lead	7439-92-1	208
Magnesium	7439-95-4	25
Manganese	7439-96-5	55
Molybdenum	7439-98-7	98
Nickel	7440-02-0	60
Phosphorous[1,2]	7723-14-0	
Potassium	7440-09-7	39
Selenium	7782-49-2	82
Silicon[1]	7740-21-3	
Silver	7440-22-4	107
Sodium	7440-23-5	23
Strontium[1,2]	7740-24-6	
Thallium	7440-28-0	205
Tin[1]	7740-31-5	118
Titanium[1,2]	7740-32-6	
Vanadium	7440-62-2	51
Zinc	7440-66-6	66

1: Additional elements performed by the laboratory but not listed in SW-846 Method 3050
2: Denotes elements not analyzed by ICP-MS

Table 2: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Matrix Spike & LCS Standard Solutions

ICP-AES Matrix Spike

Stock Standard	Concentration (mg/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)	Concentration Digestate (mg/L)
Aluminum	10000	10	500	200	2.0
Antimony	1000	25		50	0.50
Arsenic	1000	2.0		4.0	0.04
Barium	1000	100		200	2.0
Beryllium	1000	2.5		5.0	0.05
Boron	1000	25		50	0.50
Cadmium	1000	2.5		5.0	0.05
Chromium	1000	10		20	0.20
Cobalt	1000	25		50	0.50
Copper	1000	12.5		25	0.25
Iron	10000	5.0		100	1.0
Lead	1000	1.0		2	0.02
Manganese	1000	25		50	0.50
Molybdenum	1000	25		50	0.50
Nickel	1000	25		50	0.50
Phosphorous	1000	25		50	0.50
Selenium	1000	0.5		1.0	0.01
Silicon	1000	25		50	0.50
Silver	1000	2.5		5.0	0.05
Strontium	1000	25		50	0.50
Thallium	1000	2.5		5	0.05
Tin	1000	25		50	0.50
Titanium	1000	25		50	0.50
Vanadium	1000	25		50	0.50
Zinc	1000	25		50	0.50

Solution: 5% HNO_3 and 2% HCl

ICP-MS Matrix Spike

Stock Standard	Concentration (mg/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)	Concentration Digestate (mg/L)
Aluminum	10000	2.0	500	40	0.2
Antimony	1000	10		20	0.1
Arsenic	1000	1.0		2	0.01
Barium	1000	50.0		100	0.5
Beryllium	1000	1.0		2	0.01
Boron	1000	10.0		20	0.1
Cadmium	1000	1.0		2	0.01
Calcium	10000	50.0		1000	5
Chromium	1000	2.0		4	0.02
Cobalt	1000	5.0		10	0.05
Copper	1000	10.0		20	0.1
Iron	10000	10.0		200	1
Lead	1000	1.0		2	0.01
Magnesium	10000	50.0		1000	5
Manganese	1000	2.0		4	0.02
Molybdenum	1000	10.0		20	0.1
Nickel	1000	10.0		20	0.1
Potassium	10000	50.0		1000	5
Selenium	1000	1.0		2	0.01
Silver	1000	1.0		2	0.01
Sodium	10000	50.0		1000	5
Thallium	1000	1.0		2	0.01
Vanadium	1000	2.0		4	0.02
Zinc	1000	10.0		20	0.1

Solution: 2% HNO_3

ICP-AES LCS Spike

Stock Standard	Concentration (mg/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)	Concentration Digestate (mg/L)
ICP-AES Matrix Spike					
Aluminum	10000	10	500	200	2.0
Antimony	1000	25		50	0.50
Arsenic*	1000	2.0		4.0	0.04
Barium	1000	100		200	2.0
Beryllium	1000	2.5		5.0	0.05
Boron	1000	25		50	0.50
Cadmium*	1000	2.5		5.0	0.05
Chromium	1000	10		20	0.20
Cobalt	1000	25		50	0.50
Copper	1000	12.5		25	0.25
Iron	10000	5.0		100	1.0
Lead*	1000	1.0		2	0.02
Manganese	1000	25		50	0.50
Molybdenum	1000	25		50	0.50
Nickel	1000	25		50	0.50
Phosphorous	1000	25		50	0.50
Selenium*	1000	0.5		1.0	0.01
Silicon	1000	25		50	0.50
Silver*	1000	2.5		5.0	0.05
Strontium	1000	25		50	0.50
Thallium*	1000	2.5		5	0.05
Tin	1000	25		50	0.50
Titanium	1000	25		50	0.50
Vanadium	1000	25		50	0.50
Zinc	1000	25		50	0.50
Supplemental Solid LCS					
Calcium	10000	100	500	2000	20
Magnesium	10000	100		2000	20
Sodium	10000	100		2000	20
Potassium	10000	100		2000	20
Arsenic*	1000	10		20	0.2
Cadium*	1000	10		20	0.2
Lead*	1000	10		20	0.2
Selenium*	1000	10		20	0.2
Thallium*	1000	10		20	0.2
Silver*	1000	10		20	0.2

Solution: 5% HNO_3 and 2% HCl
*Elements present in multiple intermediate solutions

ICP-MS LCS Spike

Stock Standard	Concentration (mg/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)	Concentration Digestate (mg/L)
Aluminum	10000	1.0	200	50	0.5
Antimony	1000	5.0		25	0.25
Arsenic	1000	0.5		2.5	0.025
Barium	1000	25.0		125	1.25
Beryllium	1000	0.5		2.5	0.025
Boron	1000	5.0		25	0.25
Cadmium	1000	0.5		2.5	0.025
Calcium	10000	25.0		1250	12.5
Chromium	1000	1.0		5	0.05
Cobalt	1000	2.5		125	1.25
Copper	1000	5.0		25	0.025
Iron	10000	5.0		250	2.5
Lead	1000	0.5		2.5	0.025
Magnesium	10000	25.0		1250	12.5
Manganese	1000	1.0		5	0.05
Molybdenum	5000	5.0		25	0.25
Nickel	5000	5.0		25	0.25
Potassium	10000	25.0		1250	12.5
Selenium	1000	0.5		2.5	0.025
Silver	1000	0.5		2.5	0.025
Sodium	10000	25.0		1250	12.5
Thallium	1000	0.5		2.5	0.025
Vanadium	1000	1.0		5	0.05
Zinc	1000	5.0		25	0.025

Solution: 2% HNO_3

Appendix B: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Summary of changes made to this SOP;

- Added more technical detail to items in section 6.0
- Added more technical detail to items in section 7.0
- Added waste stream information

SEVERN TRENT STL

STANDARD OPERATING PROCEDURE
ACID DIGESTION OF WATERS FOR TOTAL METALS
SW-846 3010

Applicable Matrix: Water

APPROVAL SIGNATURES

Laboratory Director:	Michael F. Wheeler, Ph.D.	Date: 5/9/05
QA Manager:	Kirstin L. McCracken	Date: 5/9/05
Department Manager:	William S. Cicero	Date: 5/9/05

Proprietary Information Statement:

1.0 SCOPE AND APPLICATION

1.1. This SOP describes the laboratory procedure for the preparation of aqueous samples, EP and TCLP extracts for the determination of total metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) or Inductively Coupled Atomic Plasma-Mass Spectrometry (ICP-MS). Samples prepared by this procedure may be analyzed for the list of metals given in Table 1, Section 17.0. This digestion procedure is not applicable for the determination of total recoverable or dissolved metals.

2.0 SUMMARY OF METHOD

2.1. ICP-MS: A sample is mixed with nitric acid and refluxed with additional portions of nitric acid until the digestate is light in color or until the color has stabilized. After digestion is complete the digestate is diluted with reagent water to a final volume of 100 mL

2.2. ICP-AES: A sample is mixed with nitric acid and refluxed with additional portions of nitric acid until the digestate is light in color or until the color has stabilized. The digestate is reduced to a low volume and then refluxed with hydrochloric acid. After digestion is complete the digestate is diluted with reagent water to a final volume of 100 mL

2.3. This procedure is based on Method 3010A Test methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition September 1986.

3.0 DEFINITIONS

3.1. Definitions for general laboratory terms are included in Appendix B.

4.0 INTERFERENCES

4.1. The metals digestion area should be free of dust, particulates, and materials from fume hood because these can contribute to contamination.

4.2. The data user should be cautioned that this digestion procedure may not be sufficiently vigorous to destroy some metal complexes and precipitation in the digestate may result in a low biased silver concentration.

5.0 SAFETY

5.1. Employees must trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Specific Safety Concerns & Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. Primary Materials Used

Table 2, Section 17.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1. Block Digester: Environmental Express, 25 Position Digester or equivalent. Capable of maintaining temperature of 90-95°C

6.2. Polyethylene Digestion Vessel (100 mL) ; purchased from Environmental Express

6.3. Volumetric Pipettes (Finpipette Adjustable Pipettes; Sizes 0.10-1.00 mL & 1.00-5.00 mL or equivalent.

6.4. Class A Volumetric Flasks; 50, 100, 500, and 1000 mL sizes

6.5. Polyethyelene Ribbed Watch Glass

6.6. Whatman Filter Paper, No. 42 or equivalent

7.0 REAGENTS AND STANDARDS

7.1. Reagents

Reagent Water

Nitric Acid (HNO_3); concentrated, reagent grade; J.T. Baker or equivalent

Hydrochloric Acid (HCl); concentrated, reagent grade; J.T. Baker or equivalent

<u>1:1 Hydrochloric Acid:</u> Fill a container half full with reagent water and slowly add an equal volume of concentrated HCl. Prepare as needed.

7.2. Standards

The primary source standards are purchased from SPEX. The second source standards are purchased from Inorganic Ventures. Store the standards at room temperature and use until manufacturers assigned expiration date.

Prepare working standard solutions as needed, every 6 months or in accordance with the expiration date of the parent standard, whichever occurs first.

Matrix Spike Solution: Prepare a mixed element solution by diluting known volumes of certified stock standard solutions in reagent water. The components, recipe and final concentration in digestate for this standard are provided in Appendix A.

Aqueous Laboratory Control Sample (LCS) Solution: Prepare a mixed element solution is by diluting known volumes of prepared stock standard solutions in reagent water. The components, recipe and final concentration in digestate are provided in Appendix A.

8.0 SAMPLE HANDLING, PRESERVATION

8.1. A minimum sample volume of 500 mL should be collected in glass or polyethylene containers and immediately following collection the sample must be preserved to a pH<2 with nitric acid.

Note: Sample pH is checked on receipt in the laboratory. If the sample pH is >2, the laboratory will adjust the pH to <2. After preservation the samples must be held for a minimum of 16 hours prior to digestion.

8.2. The holding time for preserved samples is 180 days from date of collection.

8.3. Unless otherwise specified by client or regulatory program, after digestion and analysis, samples are held for a minimum of 30 days and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1. A method blank and lab control sample (LCS) must be performed with each digestion batch of 20 or fewer samples. A matrix spike (MS) should be performed with each digestion batch when sufficient sample volume is available. Sample duplicates (MD) are performed at the frequency specified by the client but a frequency of 5% of project samples is recommended.

9.2. The LCS and MS must be fortified with spike solution prior to digestion. Whenever possible, the spike solution should include all elements of interest to the project or contract.

9.3. The criteria used to assess quality control samples are given in the analytical SOP for the determinative method.

10.0 CALIBRATION AND STANDARDIZATION

10.1. Calibrate autopipettes on day of use prior to use following laboratory SOP LP-LB-0008 *Calibration of Autopipettes.* Record the calibration check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1. ICP-AES Sample Preparation

Transfer 100 mL of well-mixed sample to a polyethylene digestion vessel. Repeat for each sample and sample duplicate. Use 100 mL of reagent water for the method blank.

To prepare the LCS, transfer 50 mL of the LCS working solution to a polyethylene digestion vessel and dilute to 100 mL with reagent water. To prepare the matrix spike, transfer 100 mL of sample to a polyethylene digestion vessel and add 1 mL of the matrix spike solution.

Add 3 mL of concentrated HNO_3 to each digestion vessel, cover with a ribbed watch glass and place the vessels in the block digestor. Heat the sample at 90° to 95°C until the apparent volume has been reduced to a low volume (5 mL). Use caution to ensure the samples do not boil. Cool the beaker and add another 3 mL portion of concentrated nitric acid, cover with a non-ribbed watchglass and heat until a gentle reflux is achieved. Continue heating with additional portions of acid as needed until the digestate is light in color or does not change in appearance with continued refluxing. Evaporate to a low volume (3 mL) but do not allow any portion of the bottom of the digestion vessel to go dry. Cool the digestate. Add10 mL 1:1 HCl to each digestion vessel, cover with a watch glass and return the vessels to the block digestor. Reflux for an additional 15 minutes.

Wash down the vessel walls and watch glass with reagent water and if necessary, filter the sample remove insoluble material that could clog the nebulizer. If filtration is performed, ensure the filter apparatus and filter are thoroughly cleaned and rinse with dilute nitric acid prior to use. Adjust the final volume to 100 mL with reagent water in preparation for analysis.

11.2. ICP-MS Sample Preparation

Transfer 100 mL of well-mixed sample to a polyethylene digestion vessel. Repeat for each sample and sample duplicate. Use 100 mL of reagent water for the method blank.

To prepare the LCS, transfer 1 mL of the LCS working solution to a polyethylene digestion vessel and dilute to 100 mL with reagent water. To prepare the matrix spike, transfer 100 mL of sample to a polyethylene digestion vessel and add 0.5 mL of the matrix spike solution.

Add 3 mL of concentrated HNO_3 to each digestion vessel, cover with a watch glass and place the vessels in the block digestor. Heat the sample at 90° to 95°C until the apparent volume has been reduced to volume (5 mL). Use caution to ensure the samples do not boil. Cool the beaker and add another 3 mL portion of concentrated nitric acid, cover with a non-ribbed watchglass and heat until a gentle reflux is achieved. Continue heating with additional portions of acid as needed until the digestate is light in color or does not change in appearance with continued refluxing. Evaporate to a low volume (3 mL) but do not allow any portion of the bottom of the digestion vessel to go dry. Cool the digestate.

Wash down the vessel walls and watch glass with reagent water and if necessary, filter the sample remove insoluble material that could clog the nebulizer. If filtration is performed, ensure the filter apparatus and filter are thoroughly cleaned and rinse with dilute nitric acid prior to use. Adjust the final volume to 100 mL with reagent water in preparation for analysis.

12.0 CALCULATIONS

Not Applicable

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1. The digestion log is completed by the analyst(s) that performed the procedure and reviewed for completeness by the department supervisor or a secondary data reviewer. Problems encountered during the digestion process are documented on the digestion log or with a nonconformance report and the situation is described in the case narrative provided with the data package report.

14.0 METHOD PERFORMANCE

14.1. A demonstration of analyst capability (IDOC) is required prior to use of this SOP and any time there is a significant change in instrument type, personnel or test method. IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training.*

14.2. A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009, *Method Detection Limits & Instrument Detection Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 Waste Streams generated by this method:

- Acidic Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

16.1. Method 3010A, Test methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition September 1986.

17.0 TABLES, DIAGRAMS & FLOWCHARTS

17.1. Table 1: Target Element List
17.2. Table 2: Primary Materials Used
17.3 Appendix A: Standard Preparation

Table 1: Target Metal List

Metal	CAS #	ICP-MS isotopes
Aluminum	7429-90-5	27
Antimony	7440-36-0	123
Arsenic	7440-38-2	75
Barium	7440-39-3	135
Beryllium	7440-41-7	9
Boron[1]	7440-42-8	11
Cadmium	7440-43-9	111
Calcium	7440-70-2	44
Chromium	7440-47-3	52
Cobalt	7440-48-4	59
Copper	7440-50-8	65
Iron	7439-89-6	54
Lead	7439-92-1	208
Magnesium	7439-95-4	25
Manganese	7439-96-5	55
Molybdenum	7439-98-7	98
Nickel	7440-02-0	60
Phosphorous[1,2]	7723-14-0	NA
Potassium	7440-09-7	39
Selenium	7782-49-2	82
Silicon[1]	7740-21-3	NA
Silver	7440-22-4	107
Sodium	7440-23-5	23
Strontium[1,2]	7740-24-6	NA
Thallium	7440-28-0	205
Tin[1]	7740-31-5	118
Titanium[1,2]	7740-32-6	NA
Vanadium	7440-62-2	51
Zinc	7440-66-6	66

1: Additional elements that may be analyzed for that are not included in SW-846 Method 3010.
2: Denotes elements that are not analyzed by ICP-MS

Table 2: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Matrix Spike & LCS Standard Solutions

ICP-AES Matrix Spike

Stock Standard	Concentration (mg/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)	Concentration Digestate (mg/L)
Aluminum	10000	10	500	200	2.0
Antimony	1000	25		50	0.50
Arsenic	1000	2.0		4.0	0.04
Barium	1000	100		200	2.0
Beryllium	1000	2.5		5.0	0.05
Boron	1000	25		50	0.50
Cadmium	1000	2.5		5.0	0.05
Chromium	1000	10		20	0.20
Cobalt	1000	25		50	0.50
Copper	1000	12.5		25	0.25
Iron	10000	5.0		100	1.0
Lead	1000	1.0		2	0.02
Manganese	1000	25		50	0.50
Molybdenum	1000	25		50	0.50
Nickel	1000	25		50	0.50
Phosphorous	1000	25		50	0.50
Selenium	1000	0.5		1.0	0.01
Silicon	1000	25		50	0.50
Silver	1000	2.5		5.0	0.05
Strontium	1000	25		50	0.50
Thallium	1000	2.5		5	0.05
Tin	1000	25		50	0.50
Titanium	1000	25		50	0.50
Vanadium	1000	25		50	0.50
Zinc	1000	25		50	0.50

Solution: 5% HNO_3 and 2% HCl

ICP-MS Matrix Spike

Stock Standard	Concentration (mg/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)	Concentration Digestate (mg/L)
Aluminum	10000	2.0	500	40	0.2
Antimony	1000	10		20	0.1
Arsenic	1000	1.0		2	0.01
Barium	1000	50.0		100	0.5
Beryllium	1000	1.0		2	0.01
Boron	1000	10.0		20	0.1
Cadmium	1000	1.0		2	0.01
Calcium	10000	50.0		1000	5
Chromium	1000	2.0		4	0.02
Cobalt	1000	5.0		10	0.05
Copper	1000	10.0		20	0.1
Iron	10000	10.0		200	1
Lead	1000	1.0		2	0.01
Magnesium	10000	50.0		1000	5
Manganese	1000	2.0		4	0.02
Molybdenum	1000	10.0		20	0.1
Nickel	1000	10.0		20	0.1
Potassium	10000	50.0		1000	5
Selenium	1000	1.0		2	0.01
Silver	1000	1.0		2	0.01
Sodium	10000	50.0		1000	5
Thallium	1000	1.0		2	0.01
Vanadium	1000	2.0		4	0.02
Zinc	1000	10.0		20	0.1

Solution: 2% HNO_3

ICP-AES LCS Spike

Stock/Intermediate Standard	Concentration (mg/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)	Concentration Digestate (mg/L)
Antimony	1000	8.0	2000	4.0	2.0
*Arsenic	1000	4.0		2.0	1.0
Tin	1000	4.0		2.0	1.0
Molybdenum	1000	4.0		2.0	1.0
*Selenium	1000	2.0		1.0	0.5
*Thallium	1000	2.0		1.0	0.5
*Strontium	1000	4.0		2.0	1.0
Silicon	1000	4.0		2.0	1.0
Phosphorus	1000	4.0		2.0	1.0
Titanium	1000	4.0		2.0	1.0
AT-2					
*Aluminum	100	40		2.0	1.0
*Lead	100			2.0	1.0
Barium	50			1.0	0.5
Beryllium	50			1.0	0.5
Boron	50			1.0	0.5
*Cadmium	50			1.0	0.5
Chromium	50			1.0	0.5
Cobalt	50			1.0	0.5
*Iron	50			1.0	0.5
Manganese	50			1.0	0.5
Nickel	50			1.0	0.5
Silver	50			1.0	0.5
*Strontium	50			1.0	0.5
Vanadium	50			1.0	0.5
Zinc	50			1.0	0.5
AT-3					
*Aluminum	500	400		100	50
Calcium	500			100	50
*Iron	500			100	50
Magnesium	500			100	50
Potassium	500			100	50
Sodium	500			100	50
LCSWF					
*Arsenic	10	20		0.1	0.05
*Thallium	10			0.1	0.05
*Cadmium	5			0.05	0.025
*Selenium	5			0.03	0.015
*Lead	3			0.03	0.015

Solution: 5% HNO_3 and 2% HCl

*Elements present in multiple intermediate solutions

ICP-MS LCS Spike

Stock Standard	Concentration (mg/L)	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)	Concentration Digestate (mg/L)
Aluminum	10000	1.0	200	50	0.5
Antimony	1000	5.0		25	0.25
Arsenic	1000	0.5		2.5	0.025
Barium	1000	25.0		125	1.25
Beryllium	1000	0.5		2.5	0.025
Boron	1000	5.0		25	0.25
Cadmium	1000	0.5		2.5	0.025
Calcium	10000	25.0		1250	12.5
Chromium	1000	1.0		5	0.05
Cobalt	1000	2.5		125	1.25
Copper	1000	5.0		25	0.025
Iron	10000	5.0		250	2.5
Lead	1000	0.5		2.5	0.025
Magnesium	10000	25.0		1250	12.5
Manganese	1000	1.0		5	0.05
Molybdenum	5000	5.0		25	0.25
Nickel	5000	5.0		25	0.25
Potassium	10000	25.0		1250	12.5
Selenium	1000	0.5		2.5	0.025
Silver	1000	0.5		2.5	0.025
Sodium	10000	25.0		1250	12.5
Thallium	1000	0.5		2.5	0.025
Vanadium	1000	1.0		5	0.05
Zinc	1000	5.0		25	0.025

Solution: 2% HNO_3

Appendix B: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Summary of changes made to this SOP;

- Added more technical detail to items in section 6.0
- Added more technical detail to items in section 7.0

SOP CHANGE -IN-PROGRESS ATTACHMENT (CIPA)

SOP Title: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY
SOP No: LM-MI-6010B
Revision: 8
Date Effective: 08/05/05
CIPA Date Effective: 02/06/06

Change Approved By:

QA Manager: Kirstin McCracken
Kirstin McCracken **Date:** January 11, 2006

Inorganic Manager: William S. Cicero
William S. Cicero **Date:** January 11, 2006

The following revisions or additions in BOLD TEXT were made to the referenced SOP. These changes were implemented on the CIPA Date Effective indicated above.

Page 4 of 22: Add the following item:

6.4 Teflon Chips, use as blank soil matrix.

Page 6 of 22: Revise the following section:

10.1 Profile the instrument by aspirating a 5 ppm solution of As and running the automatic profile routine. Adjust the spectrum shifter dial on the front of the instrument and re-run the profile routine until the peak position for As is within ±0.05 units from zero. **Perform instrument maintenance when the intensity counts of the profile solution suddenly drop more than 2000 counts or when the total intensity drops by 5000.**

Page 6 of 22: Add the following section:

10.4 Troubleshooting: The following items can be checked in case of calibration failures:

Check the profile intensity with the analysis of a 5 ppm As profile solution. If the intensity has dropped by more than 2000 counts, remove and clean the torch. If the torch shows extreme signs of wear such as cracks, replace it. Change the peristaltic pump tubing and replace the transition tubing pieces (for example, those that connect the nebulizer to the mixing coil, if the tubing appears cloudy or discolored. Recheck the intensity of the profile solution to determine if maintenance performed was sufficient to correct the problem.

Page 9 of 22: Revised the following section:

14.2 Method Detection Limit (MDL) Study is required during initial method set-up and subsequently once per 12 month period **following the procedures given in the laboratory SOP for the determination of MDLs.** Instrument Detection Limit (IDL) should be determined ~~**every 6 months.**~~ **every 3 months following the procedure given in laboratory SOP for the determination of IDLs. To comply with the requirement specified in the Department of Defense (DoD) Quality System Manual, IDL values must be less than or equal to the established MDL.**

Page 12 of 22 : Revise Table 3

Table 3: QC Frequency, Criteria and Recommended Corrective Action (ICP-AES)

QC Check	Acronym	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	ICAL	Daily	NA	NA
Initial Calibration Verification	ICV	After each calibration, prior to sample analysis.	±10% of expected value %RSD between replicate integrations <5%	Correct problem, verify second source standard. If that fails, repeat calibration.
Initial Calibration Blank	ICB	Beginning of analytical sequence after ICV	No analytes ≥ RL DoD: No analytes **2X MDL**	Correct problem and reanalyze
Continuing Calibration Verification	CCV	After every 10 samples and at the end of the analytical sequence	±10% of expected value %RSD between replicate integrations <5%	Correct problem, reanalyze CCV. If that fails, repeat calibration and reanalyze all samples since last successful calibration.
Calibration Blank	CCB	Beginning of sample run, after every 10 samples and at end of the sequence (i.e. after each CCV)	No analytes ≥ RL DoD: No analytes **2X MDL**	Correct problem and reanalyze the calibration blank and previous 10 samples.
Interference Check Solutions	ICSA ICSAB	At the beginning of the analytical run	±20% of expected value	Stop analysis, locate and correct problem, reanalyze ICS and all associated QC and samples.
Low Level Standard	CRI	Daily, after ICSA and ICSAB	See Table 4 **DoD: ±30% of expected value**	Examine project DQO's. If necessary, reanalyze.
Method Blank	MB	One per digestion batch	No analytes ≥RL DoD: No analytes > ½ RL	Correct problem, redigest and reanalyze MB and associated samples.
Laboratory Control Sample	LCS	One per digestion batch *A duplicate LCS (LCSD) should be performed only per client request.*	%R= 80-120	Correct problem, redigest and reanalyze LCS, MB and associated samples for failed analytes if sufficient sample volume is available.
Matrix Spike	MS	One per batch of twenty samples or less	%R= 80-120	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Matrix Spike Duplicate	MSD	One per batch of twenty samples or less (Arizona samples only)	%R= 80-120	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Sample Duplicate	SD	One per batch of twenty samples or less	RPD ≤ 20	Examine project DQO's with Project Manager. Evaluate data to determine source of difference between results
Serial Dilution		Each digestion batch	5X dilution within ±10% of original sample result	Perform Post Digestion Spike Flag Data
Post Digestion Spike		When dilution test fails or analyte concentration in all samples <25 x MDL	%R within 75-125	Flag data

SOP CHANGE -IN-PROGRESS ATTACHMENT (CIPA)

SOP Title: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY
SOP No: LM-MI-6010B
Revision: 8
Date Effective: 08/05/05

CIPA Date Effective: 12/20/05

Change Approved By:

QA Manager: Kirstin McCracken
Kirstin McCracken
Date: December 20, 2005

Inorganic Manager: William S. Cicero
William S. Cicero
Date: December 20, 2005

The following revisions or additions in BOLD TEXT were made to the referenced SOP. These changes were implemented on the CIPA Date Effective indicated above.

Page 6 of 22: Add the following section:

10.3 Internal Standard Evaluation

Yttrium is used as an internal standard to compensate for matrix interferences. Check the response of the internal standard in every field and QC sample. The raw average should not vary by ± 30% from the raw average result of the ICB. If the IS response is outside this range, dilute and reanalyze the sample.

STANDARD OPERATING PROCEDURE
INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY
Applicable Matrices: Water/Soil/Sediment/Sludge
Standard Compound List and Reporting Limits: See Table 1

APPROVAL SIGNATURES

Laboratory Director:	Christopher A. Ouellette	Date: August 5, 2005
QA Manager:	Kirstin L. McCracken	Date: August 5, 2005
Department Manager:	William S. Cicero	Date: August 5, 2005

Proprietary Information Statement:

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure used to determine trace elements and metals in solution derived from groundwater, TCLP and EP extracts, industrial and organic wastes, sediments and other solid waste samples that have been acid digested following the procedures given in the laboratory SOPs LM-MP-3050, LM-MP-3005, or LM-MP-3010. This SOP is applicable to the determination of total recoverable metals, dissolved metals and total metals.

1.2 The elements for which this procedure is applicable are given in Table 1 along with the routine reporting limit (RL). Elements and matrices other than those listed in Table 1 may be analyzed by this procedure upon client request if performance at the concentration levels of interest is demonstrated.

2.0 SUMMARY OF METHOD

2.1 Samples are acid digested using the appropriate procedure given in the laboratory SOP LM-MP-3050, LM-MP-3005, or LM-MP-3010. The digested samples are introduced to the ICP-AES, which measures characteristic emission spectra by optical spectrometry. An aliquot of sample is nebulized and the resulting aerosol is transported to a plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometric and the intensities of the emission lines are monitored by photosensitive devices. Background correction is performed with the background measured adjacent to analyte lines on samples during analysis. The sample is analyzed by multiple integrations (2) and the average integration is converted to a concentration from a calibration curve.

2.2. The procedure is based on Method 6010B Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 2, December 1996.

3.0 DEFINITIONS

3.1. Total Recoverable Metals: The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid (Method 3005).

3.2. Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45 µm filter (Method 3005).

3.3 Total Metals: The concentration of metals determined in a sample following digestion by Methods 3010 or 3050.

3.4. Definitions for general laboratory terms are included in Appendix B.

4.0 INTERFERENCES

4.1. Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light form the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra. These effects are compensated by using computer correction of the raw data by monitoring and measurement of the interfering element and/or background correction adjacent to the analyte line.

4.2. Physical interferences are effects associated with sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies especially in samples that contain high dissolved solids and/or acid concentrations. The use of a peristalitic pump or sample dilution should minimize these interferences.

4.3. Chemical interferences such as molecular compound formation, ionization effects and solute vaporization effects are highly dependent on matrix type and specific analyte elements. These interferences are not typical with ICP-AES analysis but if observed, can be minimized by matrix matching, buffering the sample and careful selection of instrument operating conditions.

4.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample.

5.0 SAFETY

5.1. Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision, avoid looking directly at the plasma.

5.3. Primary Materials Used

Table 2, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure*.* A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 Inductively Coupled Argon Plasma Atomic Emission Spectrometer (ICP-AES); Thermo Jarrell-Ash Trace ICP-AES 61 or 61E.

6.2 Volumetric Pipettes, Size 0.10-1.00 mL & 1.00-5.00 mL ; Finpipette.

6.3 Volumetric Flasks, Class A, Size 50, 100, 500, and 1000 mL.

The vendors listed in this section are recommended and they are subject to change at the laboratory's discretion.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Hydrochloric Acid (HCl); concentrated, reagent grade; J.T. Baker.

Nitric Acid (HNO_3); concentrated, reagent grade; J.T. Baker.

7.2 Standards

Stock standard solutions are purchased from commercial vendors and stored according to the manufacturer's recommendation. Intermediate and working standard solutions are prepared as needed and unless otherwise noted, they are assigned an expiration date of 6 months from date of preparation unless the parent standard expires sooner, in which case, the earliest expiration date is used. The recommended formulations for standards used in this procedure are provided in Appendix A.

The vendors listed in this section are recommended and they are subject to change at the laboratory's discretion.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT & STORAGE

8.1 Samples may be collected in either glass or plastic containers. The sample volumes required depend on the digestion procedure but the laboratory recommends that a minimum sample volume of 500 mL be used for water samples and for soils, 5 grams. Immediately following collection, water samples must be preserved with nitric acid to a pH less than 2. If dissolved metals are to be determined, the waster sample should be filtered (on-site) prior to preservation.

8.2 The holding time is 180 days from date of collection.

8.3. Unless otherwise specified by client or regulatory program, after analysis, samples are held for 30 days and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 QC Requirements

The following QC samples are analyzed with each batch: Method Blank (MB), Laboratory Control Sample (LCS), Matrix Spike (MS) and a Serial Dilution (5X) and a sample duplicate (SD). For Arizona samples, a matrix spike and matrix spike duplicate should be performed with every batch. Sample results that exceed the linear range (high calibration standard) are diluted and reanalyzed.

In addition to analysis of the calibration standard and blank with every analytical sequence, instrument standardization is checked during the analytical run with a second source standard (ICV), Low Level Standard (CRI), Continuing Calibration Verification (CCV), Calibration Blank (CCB) and Interference Check Solutions (ICSA, ICSAB).

The minimum frequency requirements, acceptance criteria and recommended corrective action for QC samples are summarized in Table 3, Section 18.0.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Operating Conditions

Set up the instrument with the proper operating conditions using the instructions provided by the instrument manufacturer. Operating manuals for each ICP-AES instrument are located in the laboratory and specific wavelengths are listed in Table 1.

Perform plasma optimization per the manufacturer's instructions when a new instrument is set up or when there is a significant change in operating conditions in order to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array.

Establish and verify the interelement spectral interference correction routine (IECs) used during sample analysis. Verify the routine annually. To determine the IEC, analyze a single element standard for each element at 3 successive concentrations. For each element, document the presence of a positive or negative value of any other element whose absolute value exceeds the RL (interfering element). Calculate a "K" factor for each element by dividing the concentration found by the concentration of the interfering element. Take the average of the three "K" values for each interfering element and enter this value into the software system.

Determine the sensitivity (MDL), instrument detection limits (IDL), linear dynamic range and interference effects for each individual analyte line. Refer to laboratory SOP LP-LB-009 for additional guidance on the procedures for MDL and IDL studies. Determine MDLs annually and IDLs every 3 months.

Establish the upper limit of the linear dynamic range (LDR) for each wavelength used by determining the signal responses from a minimum of 2 different concentration standards across the range. One of the standards should be near the upper limit. The %R should be within ± 5% of the known value. Establish new dynamic ranges when there is a significant change in instrument response and check the range every 3 months.

Profile the instrument by aspirating a 5 ppm solution of As and running the automatic profile routine. Adjust the spectrum shifter dial on the front of the instrument and re-run the profile routine until the peak position for As is within ±0.05 units from zero.

10.2 Instrument Calibration

Calibrate the instrument daily according to the manufacturer's instructions with a calibration blank and the mixed-element calibration standard(s) following the procedure that begins in section 11.1. Immediately following analysis of the calibration standards, analyze a second source standard (ICV), calibration blank (ICB) and the continuing calibration verification (CCV) standard. Repeat a CCV and calibration blank after every tenth sample and at the end of the sequence. Analyze the ICSA and ICSAB and the low level standard (CRI) solutions after the first CCB. The criteria for the instrument check standards are provided in Section 18.0, Table 3 along with recommended corrective actions.

11.0 PROCEDURE

11.1 Standard & Sample Preparation

Transfer ~25 mL of each calibration standard [CAL #7,4,8, ICV, CCV, ICSA, ICSAB, CRI] into individual, labeled autosampler tubes. Use 25 mL of mixed acid solution (5%HCl/2%HNO3) for each calibration blank.

Transfer approximately 8 mL of each digestate to individual autosampler tubes. Prepare a serial dilution and post digestion spike using an aliquot of the un-spiked sample that was used for the matrix spike. To prepare the serial dilution, transfer 1.6 mL of parent sample to an autosampler tube and add 6.4 mL of mixed acid solution (5%HCl/2%HNO3). To prepare the post digestion spike, transfer 0.08 mL of the matrix spike solution and 7.92 mL of parent sample to an autosampler tube.

11.2 Analysis

Allow the instrument to become thermally stable prior to analysis. Create a new autosampler template on the instrument PC and enter the sample Ids in the order of analysis. Place the samples, serial dilution, post-digestion spike, calibration blanks, mixed calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis.

An example analytical sequence is given below:

Calibration Blank
Calibration Standard #7
Calibration Standard #8
Calibration Standard #4
ICV
ICB
ICSA
ICSAB
CRI
CCV
CCB
10 Samples*
CCV
CCB
10 Samples*
CCV
CCB

**The number of samples between each CCB/CCV (10) includes the method blank, laboratory control sample, matrix spike, sample duplicate, serial dilution and the post digestion spike.*

After analysis is complete, review the results against the criteria given in Section 18.0m Table 3. Perform corrective action as needed and dilute and reanalyze any sample whose result exceeds the linear calibration range.

The ICP-AES software is configured to acquire a minimum of two replicate exposures for all analyses and to use the average result of multiple exposures for standardization. The data processing software calculates results and adjusts for appropriate factors such as dilution and dry weight. Equations used are given in Section 12.0.

12.0 CALCULATIONS

12.1 Water Sample Concentration

$$C_{(ug/L)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{V_{samp}}$$

Where:
$\mu g/L_{dig}$ ICP result including dilution factors
V_{dig} Digestate Volume (mL)
V_{samp} Sample Volume (mL)

12.2 Soil/Sediment Sample Concentration

$$C_{(mg/Kg)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{g_{samp}} * \frac{100}{\%\, solids}$$

Where:
$\mu g/L_{dig}$ = ICP result including all dilution factors
V_{dig} = final digestate volume in Liters
g_{samp} = sample weight in grams

12.3 Percent Recovery (%R) LCS and CCVs

$$\%R = \frac{SR}{SA} * 100\%$$

Where:
SR= Sample Result
SA=Concentration of Spike Added

12.4 Percent Recovery (%R) MS

$$\%R = \frac{SSR - SR}{SA} * 100\%$$

Where:
SSR=Matrix Spike Result
SR=Sample Result
SA=Concentration of Spike Added

12.5 Relative Percent Difference (RPD)

$$RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} * 100$$

Where:
D1 = Sample result
D2 = Duplicate Result

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the acceptance criteria given in Section 18.0, Table 3. If the results do not fall within the established limits or criteria, perform corrective action. If corrective action is not taken or unsuccessful, record the situation and flag the data. Primary review of the data is performed by the analyst(s) that performed the procedure. Secondary review is performed by a senior analyst or a data review analyst. All data that does not meet established criteria must be flagged with the appropriate data qualifier and noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1. All analysts must perform an Initial Demonstration of Capability (IDOC) before unsupervised use of this procedure to analyze client samples. The DOC procedure is specified in the laboratory SOP for employee training.

14.2 Method Detection Limit (MDL) Study is required during initial method set-up and subsequently once per 12 month period. Instrument Detection Limit (IDL) should be determined every 6 months.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 Waste Streams generated by this method:

- Acidic Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

16.1 <u>Method 6010B Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 2, December 1996.</u> Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846).

17.0 REVISION HISTORY

17.1 Section 18.0, Table 3: The frequency for the MS/MSD/SD was updated to reflect current practice.

17.2 Section 18.0, Table 4: Control Limits for the CRI were added.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Target Analyte List, RL and Wavelength Used by Instrument
18.2 Table 2: Primary Materials Used, Hazards, Exposure Limits

18.3 Table 3: QC Frequency, Criteria and Recommended Corrective Action (ICP-AES)
18.4 Table 4: In house control charted limits for CRI standard

Table 1: Target Analyte List, Reporting Limit and Wavelengths Used by Instrument

Element	CAS No.	Reporting Limit		Wavelength	
		Water (ug/L)	Soil (mg/Kg)	TJA Trace ICP4	TJA Trace ICP6
Aluminum	7429-90-5	200	20	308.215	308.215
Antimony	7440-36-0	60	6	206.838	206.838
Arsenic	7440-38-2	10	1.0	189.042	189.042
Barium	7440-39-3	200	20	493.409	493.409
Beryllium	7440-41-7	5	0.5	313.042	313.042
Cadmium	7440-43-9	5	0.5	226.502	226.502
Calcium	7440-70-2	5000	500	317.933	317.933
Chromium	7440-47-3	10	1	267.716	267.716
Cobalt	7440-48-4	50	5	228.616	228.616
Copper	7440-50-8	25	2.5	324.754	324.753
Iron	7439-89-6	100	10	271.441	271.441
Lead	7439-92-1	3	.3	220.353	220.353
Magnesium	7439-95-4	5000	500	279.078	279.078
Manganese	7439-96-5	15	1.5	257.610	257.610
Nickel	7440-02-0	40	4	231.604	202.030
Potassium	7440-09-7	5000	500	766.491	766.491
Selenium	7782-49-2	5	.5	196.026	196.026
Silver	7440-22-4	10	1	328.068	328.068
Sodium	7440-23-5	5000	500	330.232	330.232
Thallium	7440-28-0	10	1.0	190.864	190.864
Vanadium	7440-62-2	50	5	292.402	292.402
Zinc	7440-66-6	20	2	231.856	206.200
Boron		100	10	249.678	246.678
Molybdenum		10	1	202.030	202.030
Tin		20	2	189.989	189.989
Silicon		100	10	288.158	--
Titanium		20	2	--	334.941
Strontium		20	2	--	421.552
Phosphorous		250	25	--	178.287

Table 2: Primary Materials Used, Hazards, Exposure Limits

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 3: QC Frequency, Criteria and Recommended Corrective Action (ICP-AES)

QC Check	Acronym	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	ICAL	Daily	NA	NA
Initial Calibration Verification	ICV	After each calibration, prior to sample analysis.	±10% of expected value %RSD between replicate integrations <5%	Correct problem, verify second source standard. If that fails, repeat calibration.
Initial Calibration Blank	ICB	Beginning of analytical sequence after ICV	No analytes ≥ RL DoD: No analytes ≥ MDL	Correct problem and reanalyze
Continuing Calibration Verification	CCV	After every 10 samples and at the end of the analytical sequence	±10% of expected value %RSD between replicate integrations <5%	Correct problem, reanalyze CCV. If that fails, repeat calibration and reanalyze all samples since last successful calibration.
Calibration Blank	CCB	Beginning of sample run, after every 10 samples and at end of the sequence (i.e. after each CCV)	No analytes ≥ RL DoD: No analytes ≥ MDL	Correct problem and reanalyze the calibration blank and previous 10 samples.
Interference Check Solutions	ICSA ICSAB	At the beginning of the analytical run	±20% of expected value	Stop analysis, locate and correct problem, reanalyze ICS and all associated QC and samples.
Low Level Standard	CRI	Daily, after ICSA and ICSAB	See Table 4	Examine project DQO's. If necessary, reanalyze.
Method Blank	MB	One per digestion batch	No analytes ≥RL DoD: No analytes > ½ RL	Correct problem, redigest and reanalyze MB and associated samples.
Laboratory Control Sample	LCS	One per digestion batch *A duplicate LCS (LCSD) should be performed only per client request.*	%R= 80-120	Correct problem, redigest and reanalyze LCS, MB and associated samples for failed analytes if sufficient sample volume is available.
Matrix Spike	MS	One per batch of twenty samples or less	%R= 80-120	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Matrix Spike Duplicate	MSD	One per batch of twenty samples or less (Arizona samples only)	%R= 80-120	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Sample Duplicate	SD	One per batch of twenty samples or less	RPD ≤ 20	Examine project DQO's with Project Manager. Evaluate data to determine source of difference between results
Serial Dilution		Each digestion batch	5X dilution within ±10% of original sample result	Perform Post Digestion Spike Flag Data
Post Digestion Spike		When dilution test fails or analyte concentration in all samples <25 x MDL	%R within 75-125	Flag data

Table 4: In-House Control Limits for CRI Standard

Element	Control Limit[1]
Aluminum	73-119
Antimony	76-132
Arsenic	57-149
Barium	79-119
Beryllium	72-134
Boron	83-123
Cadmium	53-145
Calcium	85-125
Chromium	52-146
Cobalt	65-127
Copper	68-139
Iron	54-190
Lead	21-191
Magnesium	83-123
Manganese	65-131
Molybdenum	67-135
Nickel	65-127
Potassium	89-129
Selenium	28-168
Silver	67-136
Sodium	76-116
Thallium	31-170
Vanadium	64-130
Zinc	65-149

[1] These control limits were derived from data generated between 11/21/04 and 01/04/05; these limits are subject to change.

Appendix A: Standard Preparation Tables

The formulations provide in this Appendix are recommended. When concentration of the component standard changes then the formulation and final concentrations must also be adjusted. Unless otherwise noted all standards are prepared in a solutions that consists of 5% Hydrochloric Acid and 2% Nitric Acid. Unless otherwise noted primary source standards are purchased from SPEX and second source standards are purchased from Inorganic Ventures.

Calibration Standard #7

Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
500 ppm Calcium	200 X-AQU-4	2000	50000
500 ppm Potassium			50000
500 ppm Sodium			50000
500 ppm Magnesium			50000
500 ppm Aluminum			50000
500 ppm Iron			50000

Calibration Standard #8

Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
1000 ppm Arsenic	1.0	2000	500
1000 ppm Selenium	1.0		500
1000 ppm Thallium	1.0		500
1000 ppm Antimony	1.0		500
1000 ppm Lead	2.0		1000
1000 ppm Tin	2.0		1000
1000 ppm Strontium	2.0		1000
1000 ppm Titanium	2.0		1000

Calibration Standard #4

Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
1000 ppm Silver	1.0	2000	500
1000 ppm Phosphorus	2.0		1000
1000 ppm Silicon	10		5000
50 ppm Beryllium	20 X-AQU-5		500
50 ppm Cadmium			500
50 ppm Strontium			500
100 ppm Antimony			1000
100 ppm Aluminum			1000
100 ppm Boron			1000
100 ppm Barium			1000
100 ppm Cobalt			1000
100 ppm Chromium			1000
100 ppm Iron			1000
100 ppm Potassium			1000
100 ppm Magnesium			1000
100 ppm Manganese			1000
100 ppm Molybdenum			1000
100 ppm Sodium			1000
100 ppm Nickel			1000
100 ppm Lead			1000
100 ppm Titainium			1000
100 ppm Vanadium			1000
100 ppm Zinc			1000
100 ppm Copper			1000

CLP-AES-CRQL Stock Standard Solution Intermediate A

Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Standard Concentration (ug/L)
100 ppb Beryllium	2 CRI-CRA-1	200	10
200 ppb Chromium			20
1000 ppb Cobalt			100
500 ppb Copper			50
300 ppb Manganese			30
800 ppb Nickel			40
200 ppb Silver			20
1000 ppb Vanadium			100
400 ppb Zinc			40

CLP-AES-CRQL Stock Standard Solution Intermediate B

Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Standard Concentration (ug/L)
600 ppb Antimony	2 CRI-CRA-2	200	120
100 ppb Arsenic	2 CRI-CRA-3		20
100 ppb Thallium			20
50 ppb Cadmium			10
50 ppb Selenium			10
30 ppb Lead			6

CRI Working Standard Solution

Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
CLP-AES-CRQL Int A	20	2000	See Above
CLP-AES-CRQL Int B	40		See Above
10000 ppm Al	0.08		400
1000 ppm Ba	0.8		400
10000 ppm Ca	2.0		10000
10000 ppm Fe	0.04		200
10000 ppm Mg	2.0		10000
10000 ppm Na	2.0		10000
10000 ppm K	2.0		10000
1000 ppm Sn	0.08		40
10000 ppm B	0.4		200
1000 ppm Mo	0.04		20
1000 ppm Sr	0.08		40
1000 ppm P	1.0		500
1000 ppm Ti	0.08		40
1000 ppm Si	0.4		200
1000 ppm Ti	0.08		40
1000 ppm Si	0.4		200

ICSA Working Standard Solution

Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
1000 ppm Fe	40	2000	200000
1000 ppm Al	100		500000
1000 ppm Ca	100		500000
1000 ppm Mg	100		500000

ICSAB Working Standard Solution

Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
1000 ppm Ag	0.4	2000	200
1000 ppm Sb	1.2		600
1000 ppm Zn	2.0		1000
1000 ppm Cd	2.0		1000
1000 ppm Ni	2.0		1000
1000 ppm As	0.2		100
1000 ppm B	3.0		1500
1000 ppm Sn	3.0		1500
10000 ppm Fe	40		200000
10000 ppm Ca	100		500000
10000 ppm Mg	100		500000
10000 ppm Al	100		500000
1000 ppm Mo	2.0		1000
1000 ppm Si	2.0		1000
1000 ppm Se	0.1		50
1000 ppm Tl	0.2		100
1000 ppm Ba	1.0		500
1000 ppm Be	1.0		500
1000 ppm Co	1.0		500
1000 ppm Cr	1.0		500
1000 ppm Cu	1.0		500
1000 ppm Mn	1.0		500
1000 ppm Pb	0.1		50
1000 ppm V	1.0		500
1000 ppm P	1.0		500
1000 ppm Ti	1.0		500
1000 ppm Sr	0.5		250

Initial Calibration Verification (ICV)

Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
100 ppm Aluminum*	10 AT-2	1000	1000
100 ppm Lead			1000
50 ppm Barium			500
50 ppm Beryllium			500
50 ppm Boron			500
50 ppm Cadmium			500
50 ppm Chromium			500
50 ppm Cobalt			500
50 ppm Iron*			500
50 ppm Manganese			500
50 ppm Nickel			500
50 ppm Silver			500
50 ppm Strontium			500
50 ppm Vanadium			500
50 ppm Zinc			500
50 ppm Copper			500
500 ppm Aluminum*	50 AT-3		25,000
500 ppm Calcium			25,000
500 ppm Iron*			25,000
500 ppm Magnesium			25,000
500 ppm Potassium			25,000
500 ppm Sodium			25,000
1000 ppm Molybdenum	0.5		500
1000 ppm Antimony	0.25		250
1000 ppm Thallium	0.25		250
1000 ppm Selenium	0.25		250
1000 ppm Arsenic	0.25		250
1000 ppm Tin	0.25		250
1000 ppm Titanium	0.5		500
1000 ppm Phosphorus	0.5		500
1000ppm Silicon	0.25		250

*Elements present in multiple intermediate solutions

CCV Working Standard Solution

Stock Standard	Stock Standard Concentration (ppm)	Volume Stock (mL)	Volume Prepared (mL)	Final Concentration (ug/L)
500 ppb Silver	Cal Standard #4	200	1000	100
1000 ppb Phosphorus				200
5000 ppb Silicon				1000
500 ppb Beryllium				100
500 ppb Cadmium				100
500 ppb Strontium*				100
1000 ppb Antimony*				200
1000 ppb Aluminum*				200
1000 ppb Boron				200
1000 ppb Barium				200
1000 ppb Cobalt				200
1000 ppb Chromium				200
1000 ppb Iron*				200
1000 ppb Potassium*				200
1000 ppb Magnesium*				200
1000 ppb Manganese				200
1000 ppb Molybdenum				200
1000 ppb Sodium*				200
1000 ppb Nickel				200
1000 ppb Lead*				200
1000 ppb Titanium*				200
1000 ppb Vanadium				200
1000 ppb Zinc				200
1000 ppb Copper				200
50000 ppb Calcium*	Cal Standard #7	600		30,00
50000 ppb Potassium*				30,00
50000 ppb Sodium*				30,00
50000 ppb Magnesium*				30,00
50000 ppb Aluminum*				30,00
50000 ppb Iron*				30,00
500 ppb Arsenic	Cal Standard #8	200		100
500 ppb Selenium				100
500 ppb Thallium				100
500 ppb Antimony				100
1000 ppb Lead*				200
1000 ppb Tin				200
1000 ppb Titanium*				200
1000 ppb Strontium				200
1000 ppm Boron		0.5		500

*Elements present in multiple intermediate solutions

Appendix B: Terms & Definitions

Analyte: The element or ion an analysis seeks to determine; the element of interest.

Analytical Sequence: The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB.

Background Correction: A technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration Blank: A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standards: A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Low Level Standard: A single parameter or multi-parameter standard solution prepared at the CRQL and used to verify the instrument calibration at low levels.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Dissolved Metals: Analyte elements in a water/aqueous sample which will pass through a 0.45 micrometer (µm) filter.

Dry Weight: The weight of a sample based on percent solids. The weight after drying in an oven.

Duplicate: A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES): A technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): solution prepared from a separate source from that which is used to prepare the calibration curve.

Interferents: Substances which affect the analysis for the element of interest.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Linear Range, Linear Dynamic Range: The concentration range over which the instrument response remains linear.

Matrix: The predominant material of which the sample to be analyzed is composed.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Effect: In general, the effect of particular matrix constituents.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a <u>range</u> where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Percent Difference (%D): The difference between the two values divided by one of the values.

Percent Solids (%S): The proportion of solid in a soil sample determined by drying an aliquot of the sample.

Preparation Blank: An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Relative Percent Difference (RPD): The relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Sample: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Serial Dilution: The dilution of a sample by a factor of five. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

SOP CHANGE -IN-PROGRESS ATTACHMENT (CIPA)

SOP Title: MERCURY (COLD VAPOR TECHNIQUE)
SOP No: LM-MI-7471
Revision: 10
Date Effective: 08/05/05
CIPA Date Effective: 02/06/06

Change Approved By:

QA Manager: Kirstin McCracken
Kirstin McCracken
Date: January 11, 2006

Inorganic Manager: William S. Cicero
William S. Cicero
Date: January 11, 2006

The following revisions or additions in BOLD TEXT were made to the referenced SOP. These changes were implemented on the CIPA Date Effective indicated above.

Page 3 of 13: Add the following item:

6.6 Teflon Chips, use as blank soil matrix.

Page 11 of 13: Revise Table 2

Table 2: QC Frequency, Criteria and Recommended Corrective Action (SW-846 7471A)

QC Check	Acronym	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	ICAL	Daily	r $\geq$ 0.995	Correct problem and repeat calibration
Initial Calibration Verification	ICV	After each calibration, prior to sample analysis.	±10% of expected value	Correct problem, verify second source standard. If that fails, repeat calibration.
Initial Calibration Blank	ICB	Beginning of analytical sequence after ICV	No analytes $\geq$RL **DoD: 2X MDL**	Correct problem and reanalyze
Low Level Standard	CRI	Per Client Request	%R (30-131) [1] **DoD: ±20% of expected value**	Correct problem, then reanalyze
Continuing Calibration Verfication	CCV	Beginning of sequence, after every 10 samples and at the end of the analytical sequence	±20% of expected value	Correct problem, reanalyze CCV. If that fails, repeat calibration and reanalyze all samples since last successful calibration.
Calibration Blank	CCB	After every 10 samples and at end of the sequence (i.e. after each IPC)	No analytes $\geq$RL **DoD: 2X MDL**	Correct problem and reanalyze the calibration blank and previous 10 samples.
Method Blank	MB	One per digestion batch	No analytes $\geq$RL DoD: ½ $\geq$RL	Correct problem, redigest and reanalyze MB and

				associated samples.
Laboratory Control Sample	LCS	One per digestion batch	%R (85-115)	Correct problem, redigest and reanalyze LCS, MB and associated samples for failed analytes if sufficient sample volume is available.
Matrix Spike	MS	One per batch of twenty samples or less	%R (85-115)	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Matrix Spike Duplicate	MSD	Per client request Arizona: 1 MS/MSD per batch	%R (85-115)	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Sample Duplicate	SD	One per batch of twenty samples or less	RPD $\leq$ 20	Examine project DQO's with Project Manager. Evaluate data to determine source of difference between results

[1] *The control limits for the low level standard (CRI) are based on control charts and are subject to change each time control charts are generated.*

STANDARD OPERATING PROCEDURE
MERCURY (COLD VAPOR TECHNIQUE)
SW-846 7471A

Applicable Matrices: Solid and Chemical Materials

APPROVAL SIGNATURES

Laboratory Director:	Christopher A. Ouellette	Date: August 5, 2005
QA Manager:	Kirstin L. McCracken	Date: August 5, 2005
Department Manager:	William S. Cicero	Date: August 5, 2005

Proprietary Information Statement:

1.0 SCOPE AND APPLICATION

1.1. This SOP describes the laboratory procedure for the determination of total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials.

1.2. The routine RL for solid samples is 0.04 mg/Kg based on a sample digestion weight of 0.3 grams and a final volume of 50 mL.

2.0 SUMMARY OF METHOD

2.1. A portion of solid sample is acid digested for 2 minutes at a temperature of 95°C then digested with potassium permanganate and potassium persulfate for 30 minutes at a temperature of 95°C. Hydroxylamine hydrochloride is added to each digestate in order to reduce excess permanganate. The digestate is placed on a closed-system mercury autoanalyzer and stannous chloride is added to each sample. The elemental mercury released is measured spectrophotometrically at a wavelength of 253.7 nm. The concentration is calculated from the response of the sample absorbance applied against the calibration curve.

2.2. This procedure is based on Method 7471A, Revision 1, September 1994. Test methods for Evaluating Solid Waste Physical/Chemical Methods (SW846).

3.0 DEFINITIONS

3.1. A list of general terms and definitions used by the laboratory is given in Appendix A.

4.0 INTERFERENCES

4.1. Potassium permanganate is added to the samples to eliminate possible interference from sulfide. Copper has also been noted as an interferent but per reference method SW-846 7471A concentrations as high as 10mg/Kg had no effect on recovery of mercury from spiked samples.

4.2. Samples high in chlorides may require additional permanganate because during the oxidation step, chlorides are converted to free chlorine which also absorbs radiation of 253nm. Care must be taken to ensure free chlorine is not present and this is accomplished by the addition of hydroxylamine hydrochloride and stannous chloride.

5.0 SAFETY

5.1. Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Protective clothing such as a lab coat, safety glasses and latex gloves must be worn while performing this procedure.

5.3. Primary Materials Used

Table 1, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure*.* A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1. Mercury Auto-Analyzer; Leeman Labs PS 200 and Leeman Labs Hydra AA with autosampler or equivalent.

6.2. Water Bath capable of maintaining temperature at 90-95°C.

6.3. Polyethylene Digestion Vessels with Volumetric Indicators; Environmental Express brand or equivalent, 100mL volume.

6.4. Volumetric Autopipettes; Finpipette brand or equivalent. Range of use 0.2-1.0mL & 1.0-5.0mL.

6.5. Top Loading Balance capable of measurements to 0.1mg.

7.0 REAGENTS AND STANDARDS

7.1. Reagents

Reagent Water

Aqua Regia: Prepare each day of use by carefully adding 3 volumes of concentrate HCl to one volume of concentrated nitric acid.

Reagent Water

Nitric Acid (HNO_3) concentrated, reagent grade.

Hydrocloric Acid (HCl), concentrated, reagent grade.

Hydroxalimine Hydrochloride, reagent grade.

Potassium Permanganate, reagent grade.

Potassium Persulfate, reagent grade.

HCl (10%): Add 100mL of concentrated HCl to a 1 L volumetric flask and adjust to 1000mL with distilled water.

Stannous Chloride Solution: Add 100 g of $SnCl_2^{\circ}H_2O$ (JT Baker or equivalent) to 1 L of 10% hydrochloric acid.

Hydroxylamine Hydrochloride: Dissolve 240 g of Hydroxylamine Hydrochloride in 2 L of reagent water.

Potassium Permanganate ($KMNO_4$): 5% solution w/v: Dissolve 100g of $KMNO_4$ in 2 L of reagent water.

Potassium Persulfate ($K_2S_2O_8$): 5% solution w/v: Dissolve 100g of $K_2S_2O_8$ in 2 L of reagent water.

7.2. Standards

Hg Stock Standard Solution (1000mg/L), Spex.

Mercury Intermediate Standard (10,000ug/L): Add 1 mL of 1000 mg/L Hg Stock Standard Solution and 0.15 mL of concentrated HNO_3 to a 100 mL volumetric flask that contains approximately 800 mL reagent water. Adjust to volume with reagent water. Assign an expiration date of six months from the date made, or the manufacturers date, whichever is sooner.

Mercury Working Standard (100 ug/L): Add 1.0 Ml of the Hg Intermediate Standard Solution and .15 Ml of concentrated HNO_3 to a 100 Ml volumetric flask that contains approximately 80 Ml reagent water. Adjust to volume with reagent water. Use this standard to prepare the calibration standards (ICAL & CCV). Prepare this standard each day of use.

ICV Stock Standard Solution (1000mg/L), Inorganic Ventures.

ICV Intermediate Standard Solution (10,000 ug/L): Add 1 mL of the 1000 mg/L ICV Stock Standard Solution and 0.15 mL of concentrated HNO_3 to a 100 mL volumetric flask that contains approximately 800 mL reagent water. Adjust to volume with reagent water. Assign an expiration date of six months from the date made, or the manufacturers date, whichever is sooner.

ICV Working Standard Solution (60 ug/L): Add 3 mL of the ICV Intermediate Standard Solution and 0.75 mL of concentrated HNO_3 into a 500 mL volumetric flask that contains approximately 300 mL reagent water. Adjust to volume with reagent water. Assign an expiration date of six months from the date made, or the manufacturers date, whichever is sooner.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1. Samples should be collected in glass or polyethylene containers. Immediately following collection the samples should be cooled to a temperature of (±2°C) and maintained at that temperature until digestion.

8.2. The holding time is 28 days from collection of the sample.

8.3. Unless otherwise specified by client or regulatory program, after digestion and analysis, samples are retained for 60 days and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1. QC Requirements

The following QC samples are analyzed with each digestion batch: Method Blank (MB) Laboratory Control Sample (LCS), a Matrix Spike (MS), and a Sample Duplicate (SD). Some client and/or regulatory programs, such as the State of Arizona, require a Matrix Spike Duplicate (MSD) instead of and/or in addition to the sample duplicate.

In addition to calibration (ICAL), instrument standardization is checked with the following QC samples, Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), and Calibration Blanks (ICB/CCB). A low level standard (CRI) is analyzed per client request. Sample results that exceed the range of calibration are diluted and reanalyzed such that the diluted sample result in near the midpoint or in the upper half of the calibration range.

The minimum frequency requirements, acceptance criteria and recommended corrective action for QC samples are summarized in Table 2, Section 18.0.

10.0 CALIBRATION AND STANDARDIZATION

10.1. Calibration

Calibrate the autoanalyzers daily with five calibration standards and a blank using the instrument operating conditions established by the instrument manufacturer. Prepare the calibration standards daily by making successive dilutions of the Hg Working Standard Solution (100ug/L) in 50 mL of reagent water. The final concentration of the prepared calibration standards is as follows:

Calibration Standards

Level	Hg Standard 100ug/L (mL)	Final Volume (mL)	Final Concentration (ug/L)
Blank	0	50	0
Level 1	0.1	50	0.2
Level 2	0.25	50	0.5
Level 3	0.5	50	1
Level 4	2.5	50	5
Level 5	5	50	10

Process the calibration standards following the procedures given in Section 11.1.The instrument data system constructs a standard curve by plotting the instrument response from each standard solution against the final concentration and using linear regression, the data system calculates the correlation coefficient. The correlation coefficient must be greater than or equal to 0.995.

10.2. Initial Calibration Verification (ICV)

Following calibration, analyze the ICV. The ICV is a second source standard whose concentration (3ppb) is near the midpoint of the calibration range but at a different concentration than the CCV (5ppb). Process the ICV following the procedure given in Section 11.1. The percent recovery of the ICV must be within 90-110%.

10.3. Continuing Calibration Verification (CCV).

Analyze a CCV initial after every 10^{th} sample and at the end of the sample run. The CCV standard is at a concentration of 5ppb and is prepared from the same source of standard used for the calibration standards. Process the CCV following the procedure given in Section 11.1. The percent recovery of the CCV must be 80-120%.

10.4. Calibration Blanks (ICB/CCB)

Analyze a calibration blank after each CCV. The results of each calibration blank must be less than the RL.

10.5. Support Equipment Calibration

Check the calibration of the auto-pipettes and the top-loading balance on the day of use prior to use and record the calibration check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1. Sample Preparation

Weigh 0.3 g of sample into a Polyethylene digestion vessel. Add 5 mL of reagent water. Use reagent water for the method blank the laboratory control sample, and each calibration blank. Add 1 mL of the Hg working standard solution (100ug/L) to the LCS and the matrix spike.

11.2. Digestion

To each sample, standard and blank add 2.5 mL of aqua regia Heat for 2 minutes in a water bath at 95°C. Allow the samples to cool then add 20mL of reagent water, 7.5 mL of potassium permanganate, and swirl to mix. Return to the hot water bath for 30 minutes. Cool and add 3 mL of hydroxylamine hydrochloride to reduce the excess permanganate. Swirl each vessel to ensure that any soluble residue dissolves back into solution. If the color of any sample is still purple, add hydroxylamine hydrochloride in 6mL increments until the purple color disappears. Add 25 mL of reagent water to each vessel and transfer the digestate to individual autoanalyzer tubes for analysis.

11.2. Instrument Set Up & Analysis

Turn the instrument lamp, gas and pump on and allow 15 minutes for the instrument to warm up. Fill the rinse bath with 10% hydrochloric acid solution. Check all tubing connections and reset the calibration curve. Check the stannous chloride reductant reservoir and fill if necessary.

Select the autosampler template and enter the sample Ids in the order of analysis. Place the samples, calibration blanks, calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis. An example analytical sequence is given below:

Example Analytical Sequence:
Calibration Blank
0.2 Calibration Standard
0.5 Calibration Standard
1.0 Calibration Standard
5.0 Calibration Standard
10.0 Calibration Standard
ICV
ICB
CRI
CCV
CCB
10 Samples*
CCV
CCB
9 Samples*
CCV
CCB

**The number of samples between each CCB/CCV (10) includes the method blank, laboratory control sample, matrix spikes, and sample duplicates.*

Select the autosampler template and enter the sample Ids into the template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin the analytical sequence. During analysis, the data processing system constructs a calibration curve by plotting the absorbances of standards versus units of mercury and sample concentrations are determined from the calibration curve.

After analysis is complete, review the data against the criteria given in Table 2, Section 18.0 and perform corrective action, as needed. Dilute and reanalyze any samples that exceed the linear range.

12.0 CALCULATIONS

12.1. Concentration

$$C_{(mg/Kg\ \ drywt.)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{g_{samp}} * \frac{100}{\%\ \ solids}$$

Where:
$\mu g/L_{dig}$ = Instrument result adjusted for dilution factors
V_{dig} = Final digestate volume
g_{samp} = Sample weight in grams
% Solids = Percent solids to nearest 0.1%

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1. Review the samples, standards and QC samples against the acceptance criteria in Table 2, Section 18.0. If the results do not fall within the established limits or criteria, corrective action. If corrective action is not taken or unsuccessful, the situation should be documented and reported in the project narrative. All data that does not meet established criteria must be noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1. A demonstration of analyst capability (IDOC) is required prior to use of this SOP and any time there is a significant change in instrument type, personnel or test method. IDOC procedures are further described in the laboratory SOP for employee training.

14.2. A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in the laboratory SOP for method detection limit studies.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1. Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

- Acid Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

16.1 Method 7471A Mercury in Solid or Semisolid Waste (Manual Cold Vapor Technique), Revision 1, September 1994. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986.

17.0 REVISION HISTORY

17.1 Section 7.1: The expiration date of the 100 ug/L working standard was changed from 6 months to daily.

17.2 Sections 10.0 and 11.0 were updated to reflect a sample volume of 50 mL instead of 100 mL.

17.3 Table 2, Section 18.0: the frequency for matrix spike was changed from per client request to a frequency of 1 each per batch of 20 or fewer samples. The acceptance criteria for the matrix spike were changed from 75-125 to 85-115 in order to be consistent with the criteria for the LCS. Control limits based on control charts were added for the low-level standard (CRI).

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: *Primary Material Used*
18.2 Table 2: *QC Summary, Acceptance Criteria, Recommended Corrective Action*

Table 1: Primary Materials Used (Mercury / CVAA)

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2: QC Frequency, Criteria and Recommended Corrective Action (SW-846 7471A)

QC Check	Acronym	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	ICAL	Daily	r $\geq$ 0.995	Correct problem and repeat calibration
Initial Calibration Verification	ICV	After each calibration, prior to sample analysis.	±10% of expected value	Correct problem, verify second source standard. If that fails, repeat calibration.
Initial Calibration Blank	ICB	Beginning of analytical sequence after ICV	No analytes $\geq$RL DoD: $\geq$MDL	Correct problem and reanalyze
Low Level Standard	CRI	Per Client Request	%R (30-131) [1]	Correct problem, then reanalyze
Continuing Calibration Verfication	CCV	Beginning of sequence, after every 10 samples and at the end of the analytical sequence	±20% of expected value	Correct problem, reanalyze CCV. If that fails, repeat calibration and reanalyze all samples since last successful calibration.
Calibration Blank	CCB	After every 10 samples and at end of the sequence (i.e. after each IPC)	No analytes $\geq$RL DoD: $\geq$MDL	Correct problem and reanalyze the calibration blank and previous 10 samples.
Method Blank	MB	One per digestion batch	No analytes $\geq$RL DoD: ½ $\geq$RL	Correct problem, redigest and reanalyze MB and associated samples.
Laboratory Control Sample	LCS	One per digestion batch	%R (85-115)	Correct problem, redigest and reanalyze LCS, MB and associated samples for failed analytes if sufficient sample volume is available.
Matrix Spike	MS	One per batch of twenty samples or less	%R (85-115)	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Matrix Spike Duplicate	MSD	Per client request Arizona: 1 MS/MSD per batch	%R (85-115)	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Sample Duplicate	SD	One per batch of twenty samples or less	RPD $\leq$ 20	Examine project DQO's with Project Manager. Evaluate data to determine source of difference between results

[1] The control limits for the low level standard (CRI) are based on control charts and are subject to change each time control charts are generated.

Appendix A: Terms & Definitions

Analyte: The element or ion an analysis seeks to determine; the element of interest.

Analytical Sequence: The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration: The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards.

Calibration Blank: A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Duplicate: A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): solution prepared from a separate source from that which is used to prepare the calibration curve.

Interferents: Substances which affect the analysis for the element of interest.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Relative Percent Difference (RPD): As used in the SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

Sample: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

STANDARD OPERATING PROCEDURE
POLYCHLORINATED BIPHENYLS (PCB'S) BY GC/ECD
SW-846 METHOD 8082
Applicable Matrices: Non-Potable Water, Solid and Chemical Materials
Standard Compound List and Reporting Limits: See Table 1

APPROVAL SIGNATURES

Laboratory Director:	Christopher A. Ouellette	Date: December 12, 2005
QA Manager:	Kirstin L. McCracken	Date: December 12, 2005
Organics Manager:	Jennifer L. Clements	Date: December 12, 2005

Proprietary Information Statement:

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the determination of concentrations of Polychlorinated Biphenyls (PCBs) in extracts derived from non-potable water, solids, tissue, air, and chemical materials including TCLP leachates, using dual column Gas Chromatography with Electron Capture Detectors (GC/ECD). This SOP is applicable to the analytical procedure only; the extraction and extract cleanup methods referenced in this SOP are described in the following laboratory SOPs:

LM-OP-3510	Separatory Funnel Extraction
LM-OP-3540	Soxhlet Extraction
LM-OP-3541	Automated Soxhlet Extraction
LM-OP-3550	Ultrasonic Extraction
LM-OP-Cleanup	Extract Cleanup Procedures
LM-OP-GPC	Gel Permeation Chromatography (GPC)

1.2 The analytes that can be determined by this procedure and their associated Reporting Limits (RL) are listed in Table 1, Section 18.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate matrix-specific extraction technique. After extraction, the extract may be subject to cleanup depending on the nature of sample matrix and the target analytes. After cleanup, the extract is analyzed by injecting a 2-µl aliquot into a dual capillary column GC/ECD.

2.2 This procedure is based on SW-846 Method 8082, Revision 0, December 1996.

3.0 DEFINITIONS

A list of terms and definitions is given in Appendix C.

4.0 INTERFERENCES

4.1 Contaminated solvents, reagents or equipment can cause interferences. To reduce the occurrence of this type of interference, glassware must be cleaned thoroughly before use following the procedure given in laboratory SOP LM-OP-Glass *Glassware Washing*. Solvents and acids are lot- tested and approved for use before delivery to the laboratory in accordance with STL-T-001 *Testing of Solvent and Acids*. Solvents should be stored in an area away from organochlorine compounds to minimize contamination.

4.2 Phthalate esters introduced during sample preparation can pose a problem in the determination of pesticides. Common flexible plastics contain varying amounts of phthalate esters, and these can be easily extracted or leached during extraction. To minimize this interference, avoid contact with any plastic materials.

4.3 Non-target compounds co-extracted from the sample matrix can also cause interference, the extent of which will vary considerably depending on the nature of the samples. Elemental sulfur is often found in sediment samples and its presence will result in broad peaks. Samples are screened before analysis and those samples that contain high levels of sulfur are subject to cleanup using activated copper before analysis (SW-846 3660B). Waxes, lipids, other high molecular weight materials and co-eluting organophosphorous pesticides may be removed by extract cleanup with GPC (SW-846-3640A). Co-eluting chlorophenols can be eliminated by cleanup with silica gel (SW-846 3630C), or Florisil (SW-846 3620B), or Sulfuric acid Cleanup (SW-846 3665A) may be used to eliminate certain organochlorine pesticides and elevated baselines.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Specific Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 Primary Materials Used

Table 2, Section 18 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. Note: The table does not include all materials used in the procedure. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used can be found in Section 7. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Autosampler Vials, National Scientific or equivalent.

6.2 Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP9000 D250 (Chemsvr4) and an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5.

6.3 GC/ECD: with dual columns, dual ECDs, and auto-sampler capable of a 2-µl injection split onto two columns: HP 5890 with Leap Technology CTC A200SE and A200S Fisons autosamplers, or equivalent.

6.4 GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses.

- RTX-5, (30m x 0.25 mmID x 0.25um)
- RTX-35, (30m x 0.25 mmID x 0.25um)
- Equivalent columns may be used, provided the elution orders are documented and compound separations are maintained.

6.5 Hydrogen Generator: Whatman.

6.6 Volumetric Syringes, Class "A" (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

- Hexane JT Baker Ultra-Resi analyzed.

7.2 Standards

Stock standard solutions are purchased made from commercial vendors. Intermediate and working standards solutions are prepared in the laboratory by diluting a known volume of stock standard in an appropriate solvent and diluting to a specified volume. Standard preparation procedures for intermediate and working standard solutions are provided in Appendix A.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Sample extracts must be stored at 4°C ± 2° until the time of analysis. The analytical holding time is 40 days from date of sample extraction.

8.2 Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 The minimum frequency requirements, acceptance criteria and recommended corrective action for all QC samples are summarized in Section 18, Table 3. Below is a summary of each type of QC sample that is analyzed with the method.

9.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each extraction batch. These samples show that the laboratory is in control, independent of the sample matrix.

9.3 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each extraction batch. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.4 A Surrogate spike is added to all field and QC samples before extraction to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.5 Instrumental QC standards include a five-point ICAL is generated for Aroclor 1016 and 1260 (referred to as AR1660) and an initial one point calibration for all other Aroclors. After the ICAL, an Initial Calibration Verification (ICV) standard, also referred to as a second source standard, containing Aroclor 1660 is analyzed to verify the ICAL standard formulation. Continuing Calibration Verification (CCV) standards are analyzed before sample analysis, every ten samples thereafter, and at the end of the run to assess instrument drift.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column to the injection port and connect the guard column to the separate analytical columns using a glass "Y". Then attach the analytical columns to the dual ECD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature:	130°C for 1 minute
Temperature Program:	20°C per minute to 190°C to 5°C per minute to 225°C to 20.0°C per minute to 300°C. Hold for 6 minutes.
Detector Temperature	310°C
Injector Temperature:	225°C
Injection volume:	2µL
Carrier Gas:	Hydrogen (supplied by hydrogen generators)

Optimize the flow rate of the carrier gas by injecting an un-retained substance onto the column at an isothermal oven state and adjusting the flow to obtain the recommended dead volume time.

10.2 Initial Calibration

Before initial or daily calibration, inject an instrument blank (IBLK) consisting of hexane blank to bring the GC/ECD system online.

A multi-point calibration of AR1660 at five concentrations is sufficient in demonstrating linearity. AR1660 includes most of the peaks represented in the other five Aroclors. For the remaining Aroclors, a midlevel standard is analyzed to aid in pattern recognition and is used as a single point calibration standard. Five point calibrations for these aroclors will only be performed when required by the client or when specified as a regulatory requirement. A minimum of three to five peaks per aroclor is used for quantificaton. The calibration standards are introduced using the same technique that is used for sample extracts described in Section 11.

Inject 2-µl of each calibration standard and calculate the Calibration Factor (CF), mean CF and Percent Relative Standard Deviation (% RSD) for each analyte on both columns (Appendix B). The %RSD for each peak must be ≤ 20 % for the curve to be considered acceptable. If any of the results are outside QC criteria, the cause of the problem is investigated and corrected prior to analysis of samples.

10.3 Alternate Quantificaton. In some cases, it may be preferable to use linear regression to quantify the compounds. The following approaches may be used:

Linear Regression - A curve of concentration vs. peak area is generated for each analyte and the correlation coefficient is calculated. The calibration must have a correlation coefficient (r) ≥ 0.99 (0.995 for DoD) for acquisition of samples to continue. The use of linear regression requires a minimum of 5 calibration points. See SW-846 Method 8000B for linear regression calculations.

Once a method of calibration is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is generated.

10.4 Retention Time Windows

When a new GC column is installed, RT windows are established for 3-5 peaks for Aroclor 1660 by analyzing three standards over a 72-hour period and calculating the mean RT and Standard Deviation (SD). The RT window is calculated as mean RT ± 3SD of the three standards. If the SD is <0.01 minutes, the laboratory may use a default SD of 0.01 minutes.

If, in the professional judgment of the analyst, this results in an RT window that is too tight and would favor false negatives, the laboratory may opt use an alternate method to determine the RT windows as follows: using the RT of the midpoint initial calibration standard, calculate the RT window using ± 0.05 minutes from the midpoint of the RT in the initial calibration.

10.5 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV (Appendix A). The calculated concentration of each analyte must be within ± 15% of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate. The acceptance criteria must be met on both columns.

10.6 Calibration Verification (CCV)

A CCV containing AR1660, at or below mid-calibration range, is analyzed each day before sample analysis, after every ten injections and at the end of each analytical batch to monitor instrument drift. The concentration of the CCV is varied. Calculate the CF and percent difference or drift (Appendix B) for each analyte on both columns. The percent difference or drift must be within ±15% for each analyte. Compare the RT of each analyte in the CCV with the RT windows; the RT must be within the window established in 10.4. The acceptance criteria must be met on both columns.

If the CCV fails, it may be repeated once. If it still fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples must be bracketed by passing CCVs, and samples before and after CCV failure must be reanalyzed, unless the CCV is high and there are no detects in the associated samples.

10.7 Troubleshooting: the following items can be checked in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if chromatography has degraded as evidenced by peak shapes.
- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column.
- Needle crushed during injection - Replace the needle.
- Auto-sampler failure - Reset the auto-sampler.
- Power failure - Reset run in Multichrom and re-acquire or re-initiate run sequence.

11.0 PROCEDURE

11.1 Transfer approximately 100-µl of each QC standard and sample extract to an autosampler vial and place the vials in the auto-sampler. Arrange the samples in a sequence that begins with the calibration standards followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs)

11.2 Arrange the samples in a sequence that begins with the calibration standards (if necessary) followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs).

An example analysis sequence is given below:

Injection Number	Lab Description
1	Instrument Blank
2	AR1221 200PPB
3	AR1232 200PPB
4	AR1242 200PPB
5	AR1248 200PPB

6	AR1254 200PPB
7	AR1660 50PPB
8	AR1660 100PPB
9	AR1660 200PPB
10	AR1660 400PPB
11	AR1660 800PPB
12	ICV
13-22	10 injections
23	CCV
	Repeat until ending with CCV

11.3 Enter the sample ID's into the data acquisition program in the order the samples were placed in the autosampler and start the analytical sequence.

11.4 Cleaning blanks (CBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst.

11.5 The data system identifies the target analytes by comparing the retention time of the peaks to the retention times of the initial calibration standards. The data system does not recognize aroclor patterns. The analyst manually identifies aroclors by comparing the pattern in the samples to the patterns in the initial calibration standards. When "weathered" aroclor patterns are present, the laboratory identifies aroclors based on the best overall pattern match. Using an average of the chosen quantificaton peaks per aroclor, the data system calculates the corrected concentration for each target analyte from the calibration curve using the equations given in Appendix B. If sample interference is suspected, the laboratory may remove up to two quantificaton peaks per column. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP LP-LB-0006 *Manual Integration.*

11.6 After analysis is complete, evaluate the results against the performance criteria given in Section 10 and Table 3, Section 18 and perform corrective action as necessary.

11.7 Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should ideally result in a determination within the upper half of the calibration curve.

12.0 CALCULATIONS

See Appendix B.

13.0 DATA ASSESSMENT, CORRECTIVE ACTION & REPORTING

13.1 Data Review and Corrective Action

Review the samples, standards and QC samples against the acceptance criteria in Table 3. If the results do not fall within the established limits, perform the recommended corrective action. If corrective action is unsuccessful, document the situation with a nonconformance report and/or qualify the data using an appropriate data qualifier (see Appendix C for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

In the absence of project specific requirements, use the control limits specified in Table 1. The control limits in Table 1 are based on in-house statistically generated limits. In some cases, the in-house limits were outside of Department of Defense (DoD) limits as specified in the Quality Systems Manual for Environmental Laboratories. Where this is the case, the laboratory uses the stricter, DoD limits that are presented in bold in Table 1. For DoD projects, the in-house laboratory limits are also included in the project report.

Weathering of PCB's in the environment may alter the PCB's to the point that the pattern no longer matches the pattern established for that Aroclor in the initial calibration. The laboratory takes the best pattern match approach to the identification and quantification of weathered PCB's. In many cases, this entails choosing peaks such that the weathering pattern does not affect the quantificaton of the Aroclor.

13.2 Data Reporting

Unless otherwise specified, the higher result between the two columns is reported. The Relative Percent Difference (RPD) of the two results is calculated, and if the RPD is greater than 40% it is reported with a data flag. If, in the analyst's judgment, the higher result is due to overlapping peaks, or interference peaks, the lower of the two results should be reported with a data flag, and the issue discussed in the project narrative.

The laboratory's RL for each target analyte is provided in Table 1. Report the data to the RL adjusted for sample matrix, percent moisture, and sample dilution/concentration. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results that have concentrations for a target analytes less than the RL with a "U" qualifier. Unless otherwise specified, report the results for solid matrices on a dry weight basis.

Some projects may require reporting positively identified target analytes less than the RL. In this case, the analyte can be qualitatively detected but not accurately quantified. Flag all results less than the RL with a "J" data qualifier (Appendix C).

Some projects may require RLs that are less than the laboratory's routine RL. Sample results may be reported to the project RL if the project RL is greater than the Quantification Limit (QL) and above the MDL. In this context, the QL is defined as the concentration of the low calibration standard. If the project RL is less than the QL, all values less than the QL must be reported as estimated and qualified with a “J”.

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-LB-009 *Determination of Method Detection Limits.*

13.3 Data Management and Records: All electronic and hardcopy data is managed, retained, and archived as specified in laboratory SOP LP-QA-0014 *Laboratory Records*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.4 The laboratory statistically derived control limits used to evaluate accuracy, precision and surrogate recoveries are provided in Table 1. The control limits for accuracy are based on compiled data and are set at 3 standard deviations around the mean using the procedures described in laboratory SOP LP-QA-012 *Control Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in Section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 The following waste streams are produced when this method is carried out.

- Waste Solvents
- Solid Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

Polychlorinated Biphenyls by Gas Chromatography (Method 8082), Revision 0; December 1996. USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

17.0 SOP REVISION HISTORY

The following changes were made in this revision:

Section 6:	Added computer hardware and software and hydrogen generator.
Section 7:	7.1 - Removed solvents not used in analytical method.
Section 10:	10.4 - Changed RT window requirements to assess RT window from initial calibration curve. 10.6 Added detail about repeating CCV. 10.7 Added Troubleshooting.
Section 13:	13.1 - Added detail regarding the use of DoD LCS and Surrogate Limits. 13.2 – Moved dual column reporting from Section 11. Changed to reporting higher value and flagging at 40%. 13.3 - Added SOP reference for Data Management & Records.
Section 17:	New Section added.
Table 1:	Updated control limits. Removed TCMX as surrogate. Added footnotes.
Table 2:	Removed solvents not used in analytical method.
Table 3:	Re-worded corrective action for MB, MS/MSD, SD. Added detail to corrective action section.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

Table 1:	Target Analyte List, Reporting Limits, and Control Limits as Accuracy (%R) and Precision (RPD)
Table 2:	Primary Materials Used
Table 3:	QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action
Appendix A:	Standard Preparation Tables
Appendix B:	Equations
Appendix C:	Terms & Definitions

Table 1: Target Analyte List, Reporting Limits[1], and Control Limits[2] as Accuracy (%R) and Precision[4] (RPD)

Analyte	Reporting Limit[1]		Water		Solid, Chemical Material, Tissue	
	Water ug/L	Soil ug/Kg	%R	RPD[4]	%R	RPD[3]
AR1016	0.50	17	55-125	≤ 35	55-120	≤ 30
AR1221	0.50	17	NA	NA	NA	NA
AR1232	0.50	17	NA	NA	NA	NA
AR1242	0.50	17	NA	NA	NA	NA
AR1248	0.50	17	NA	NA	NA	NA
AR1254	0.50	17	NA	NA	NA	NA
AR1260	0.50	17	55-120	≤ 30	60-130	≤ 35
DCB (Surrogate)	NA	NA	55-120	NA	60-125	NA

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.
[2] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.
[3] RPD for MS/MSD only.

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Correct
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CF: RSD ≤ 20% Linear Regression: r ≥ 0.99 (0.995 for DoD)	Correct problem, reanalyze, repeat ca
ICV	After each initial calibration	%Difference ± 15% from expected value	Correct problem and verify second so fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±15%	Re-analyze once, if still outside criteri action, sequence can be re-started if t pass, otherwise repeat ICAL and all a since last successful CCV, unless CC bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	< RL DoD: ≤ ½ RL If analyte in any sample ≥ RL	Examine project DQO's and take app action, which may include re-analysis batch, and/or non-conformance repor action must be documented on NCR. in samples, or if all detects are > 10 X reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	Evaluated against control limits in Table 1	Examine project DQO's and take app action, which may include re-analysis of batch, and/or non-conformance rep action must be documented on NCR. values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	Evaluated against control limits in Table 1	Evaluate data and determine if a matr error is indicated. If analytical error, re extract. Flag all reported values outsic
Surrogate Spike	All field and QC samples	Evaluated against control limits in Table 1	Evaluate data and determine if a matr error is indicated. If analytical error, re If matrix effect, review project DQOs t effect must be confirmed by re-analys values outside of control limits.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Appendix A: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP LP-LB-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

All standards are prepared using volumetric glassware, including Class A volumetric flasks, and Hamilton Syringes. Unless otherwise noted, or unless the expiration date of the parent standard is earlier, an expiration date of 6 months from date of preparation is assigned to all intermediate and working standards. If the expiration date of any of parent standards is earlier, that earlier expiration date is used.

Table Legend:
C_{stock} = Concentration of Parent Standard
V_{stock} = Volume of Parent Standard
V_{std} = Volume of Prepared Standard
C_{std} = Theoretical Concentration of Prepared Standard

INTERMEDIATE STANDARDS – in hexane

Intermediate Standards-10mg/L each: AR1660, AR1254, AR1248, AR1242, AR1232, AR1221
These are prepared as 6 individual standards in hexane.

Stock Standard	Restek Catalog #	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (mg/L)
AR1016/AR1260[1]	32039	1000	0.400	40	10
AR1254	32011	1000	0.400	40	10
AR1248	32010	1000	0.400	40	10
AR1242	32009	1000	0.400	40	10
AR1232	32008	1000	0.400	40	10
AR1221	32007	1000	0.400	40	10

[1] Concentration represents concentration of each Aroclor in mixed standard rather than concentration of both.

ICV – Second Source Standard - AR1660 10mg/L

Stock Standard	Restek Catalog #	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (mg/L)
AR1016/AR1260	32039*	1000	0.400	40	10

* ***Must*** be from a different lot than calibration standards!

WORKING STANDARDS – in hexane

DCB 10mg/L

Stock Standard	Restek Catalog #	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (mg/L)
Pesticide Surrogate	32000	1000	0.400	40	10

CALIBRATION STANDARDS – in hexane

AR1660 Calibration Level 5 - 800 μg/L

Stock Standard	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (μg/L)
AR1660 Intermediate Standard	10	8.0	100	800
DCB Working Standard	10	0.800	100	80

Prepare Calibration Standards Level 1-4 in hexane using AR1660 Calibration Level 5 as stock:

AR1660 Calibration Working Standards (Level 1-4)

AR1660 800 ppb (Calibration Level 5)	400 ppb	200 ppb	100 ppb	50 ppb
V_{stock} mL	20	25	5.0	2.5
V_{std} mL	40	100	40	40

AR1254 Working 200μg/L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1254 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1248 Working 200μg /L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1248 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1242 Working 200μg /L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1242 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1232 Working 200μg /L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1232 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1221 Working 200ug/L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1221 Intermediate	10	800	40	200
DCB Working	10	80	40	20

ICV – Second Source Standard AR1660 Working 200ug/L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1660 Intermediate	10	1000	50	200
DCB Working	10	100	50	20

Final Concentration of Prepared Calibration Standards

Analyte	Level 1 ug/L	Level 2 ug/L	Level 3 ug/L	Level 4 ug/L	Level 5 ug/L
DCB (Surrogate)	5	10	20	40	80
AR1660	50	100	200	400	800
AR1254	NA	NA	200	NA	NA
AR1248	NA	NA	200	NA	NA
AR1242	NA	NA	200	NA	NA
AR1232	NA	NA	200	NA	NA
AR1221	NA	NA	200	NA	NA

Appendix B: Equations

Calibration Factor (CF_x) = $\dfrac{\text{Peak area or height}_{(x)}}{\text{Standard concentration}_{(ug/L)}}$

Mean Calibration Factor ($\overline{CF}$) = $\dfrac{\sum_{i=1}^{n} CF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Calibration Factor (SD) = $\sqrt{\dfrac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor = $\dfrac{SD}{\overline{CF}} \times 100\%$

Percent Difference (%D) = $\dfrac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$

where: CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\dfrac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ X 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$$C_{extract}\,(\text{ug/L}) = \frac{\text{Peak Area (or Height)}}{\overline{\text{CF}}}$$

Note: The concentrations of the 3-5 peaks chosen for quantificaton is calculated and the average is then taken for final calculation.

Water

$$C_{sample}(\text{ug/L}) = C_{extract}(\text{ug/L}) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Solids

$$C_{sample}(\text{ug/Kg}) = C_{extract}(\text{ug/L}) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\text{\% solids}} \times DF$$

where: DF = Extract Dilution Factor. If no dilution was made, DF=1.

Appendix C: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

The qualifiers that are routinely used for this test method are:

U: Compound analyzed for but not detected at a concentration above the reporting limit.
J: Estimated Value
P: There is greater 40% difference for detected concentrations between two GC columns
C: Positive result whose identification has been confirmed by GC/MS
B: Compound is found in the sample and the associated method blank.
E: Compound whose concentration exceeds the upper limit of the calibration range.
D: Concentration identified from a dilution analysis.

X,Y,Z: Laboratory defined flags that may be used alone or combined as needed. If used, provide a description of the flag in the project narrative.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

GEORGIA-PACIFIC CORPORATION
KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

TIME CRITICAL REMOVAL ACTION
QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Prepared by: Blasland, Bouck & Lee, Inc. Date: ________________________

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U.S. Environmental Protection Agency Region 5

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Acronyms

BBL	Blasland, Bouck & Lee, Inc.
BBEPC	Blasland & Bouck Engineers, P.C.
CLP	Contract Laboratory Program
DQO	Data Quality Objectives
EDD	Electronic Data Deliverable
FSP	Field Sampling Plan
Georgia-Pacific	Georgia-Pacific Corporation
KRSG	Kalamazoo River Study Group
MDEQ	Michigan Department of Environmental Quality
mg/kg	milligrams per kilogram
Mill Properties	collectively, the Georgia-Pacific Kalamazoo Mill Property and the Former Hawthorne Mill Property
MS	Matrix Spike
MSB	Matrix Spike Blank
MSD	Matrix Spike Duplicate
NAAQS	National Ambient Air Quality Standards
NELAP	National Environmental Laboratory Accreditation Program
Part 201	Michigan's Part 201 Natural Resources and Environmental Protection Act
PCB	Polychlorinated Biphenyls
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
residuals	Paper-Making Residuals
RI/FS	Remedial Investigation/Feasibility Study
SOP	Standard Operating Procedure
STL	Severn Trent Laboratories, Inc.
SVOC	Semivolatile Organic Compound
TAL	Target Analyte List
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TCRA	Time-Critical Removal Action
TMP	Remedial Action Turbidity Monitoring Plan
TOC	Total Organic Carbon
TSS	Total Suspended Solids
$\mu g/m^3$	micrograms per cubic meter
USEPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound
Work Plan	*Time Critical Removal Action Work Plan*
ug/L	Microgram Per Liter

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A Laboratory NELAP Accreditation
B Laboratory Standard Operating Procedures (SOPs) – The Laboratory SOPs associated with this QAPP Addendum are on two attached CDs
C Anticipated Sampling Grids and Associated Calculations
D Particulate Monitoring SOP

1. Project Management and Objectives

This *Quality Assurance Project Plan Addendum* (QAPP Addendum) updates the QAPP (Blasland & Bouck Engineers, P.C. [BBEPC], 1993a) developed to support the *Remedial Investigation/Feasibility Study Work Plan* (RI/FS Work Plan) for the Allied Paper Portage Creek Kalamazoo River Superfund Site (BBEPC, 1993b).

This QAPP Addendum specifically identifies the protocols and methods which will be employed to assure the quality of data collected as part of the Time-Critical Removal Action (TCRA) for the removal of paper-making residuals (residuals) and soils that contain, or may contain, polychlorinated biphenyls (PCB) from the Georgia-Pacific Corporation (Georgia-Pacific) Kalamazoo Mill Property (Kalamazoo Mill Property) and the former Hawthorne Mill Property (Hawthorne Mill Property), collectively referred to as the Mill Properties (Figure 1).

The specific sampling requirements and the locations and numbers of samples to be taken, are found in the *TCRA Work Plan* (Work Plan) (BBL, 2006). The Work Plan provides the rationale for the locations and numbers of samples and the selection of measurements and chemical analytes.

The procedures specified herein will be used for the sampling and analysis of soils, residuals, ambient air, and water for PCB to determine if specified performance standard are met or action levels are exceeded. Additionally post excavation soil samples will be analyzed for other constituents to characterize the soil. Turbidity in surface water will be monitored in the Kalamazoo River at locations upstream and downstream of the excavation activities in the Refuse Area. In addition, dust generation will be monitored during TCRA construction activities that potentially may generate dust.

1.1 Project Organization

BBL maintains overall technical responsibility for the TCRA at the Mill Properties. As such, BBL will perform sampling associated with construction activities, compile and report resulting data, provide quality assurance/quality control (QA/QC) oversight, and prepare all associated reports.

The direct management of the technical and administrative aspects of the TCRA will be accomplished by representatives of Georgia-Pacific, BBL, and United States Environmental Protection Agency (USEPA) Region 5. Currently, the following personnel have been assigned to this project:

Affiliation	Title	Name	Phone #
USEPA Region 5	Remedial Project Manager	Shari L. Kolak	312-886-6151
USEPA Region 5	Quality Assurance Manager	TBD	--
BBL	Project Coordinator/Manager	Patrick N. McGuire	315-671-9233
BBL	Field Manager	TBD	--
BBL	Quality Assurance Manager	Dennis K. Capria	315-671-9299
BBL	Data Manager	Michael D. Scoville	315-671-9387

The analytical laboratory services for this project will be provided by Severn Trent Laboratories, Inc. (STL) in Burlington, Vermont. STL is accredited under the National Environmental Laboratory Accreditation Program (NELAP). A certificate of accreditation is provided in Appendix A.

Affiliation	Title	Name	Email address	Telephone #
Severn Trent Laboratories, Inc.	Laboratory Project Manager	James Madison	jmadison@stl-inc.com	802-655-1203
	Quality Assurance Officer	Kirstin McCracken	kmccracken@stl-inc.com	802-655-1203

1.2 Project Description

1.2.1 Project Overview

The scope of work for the TCRA at the Mill Properties is detailed in the Work Plan and consists of the following activities:

- Excavate residuals and soils that contain, or may potentially contain PCB concentrations exceeding performance standard of 10 mg/kg from the Refuse Area and Oxbow Area and dispose of them at the A-Site (Figure 1).

- Excavate visibly stained soil from beneath the Transformer Pad Area and dispose of it at a Type II landfill.

- Sample soil after excavation and characterize it for Target Compound/Target Analyte List (TCL/TAL) constituents.

- Excavate the pipeline and wet well at the Wastewater Pipeline Area and dispose of them at the A-Site.

- Restore the Refuse Area and Oxbow Area.

1.2.2 Project Schedule

A tentative schedule for the Mill Properties TCRA is shown on Figure 3 in the TCRA Work Plan and included in this QAPP Addendum. The schedule will be updated as necessary and reported in the monthly reports prepared for the TCRA.

1.3 Project Planning and Problem Definition

1.3.1 Project Planning Meetings

Project planning meetings and/or teleconferences have been and will continue to be scheduled as needed to develop the TCRA and monitor ongoing work activities detailed in the Work Plan. Meetings involving Georgia-Pacific and USEPA Region 5 will be coordinated through the Project Coordinator or designated representative.

1.3.2 Quality Objectives and Criteria for Measurement

The data quality objective (DQO) process, as described in *Guidance for QA Project Plans* (USEPA, 2002b), is intended to provide a "logical framework" for planning field investigations. The following section addresses, in turn, each of the seven sequential steps in the USEPA's QAPP DQO process.

Step 1: Problem Statement
The TCRA is being conducted to remove paper-making residuals and soils that contain or may potentially contain PCB from the Mill properties. As a result of TCRA activity, other matrices such as soil, residuals, ambient air, and water will be analyzed for PCB. Post excavation soil sampling will also include analysis for other for the characterization of post-excavation soil only.

Step 2: Decision Identification
The initial use of the data is descriptive (distribution and concentration). The decision in this case, will be determined by subsequent review of the data collected during the TCRA and evaluated as follows:

- Verify that PCB concentrations remaining in soil following removal activities meet the Performance Standard of 10 milligram per kilogram (mg/kg) as presented in Table 1-1A.
- Verify that PCB concentrations in treated water meet the discharge limit of 2.6 x 10^{-5} microgram per liter (ug/L) (or not detected) prior to discharge as presented in Table 1-1A.
- Perform environmental monitoring for dust and ambient air for PCB during TCRA construction activities and to verify that PCB concentrations in ambient air are at or below the action levels presented in Table 1-1B.
- Sample analysis performed for characterization for disposal as presented in Table 1-1C.

Step 3: Identifying Decision Inputs
Decision inputs incorporate both the concentration and distribution of PCB in site media. A fundamental basis for decision making is that a sufficient number of sample results of acceptable quality must be available from the TCRA post-removal verification sampling air monitoring, water monitoring, and disposal characterization to support the decision. Thus, the necessary inputs for the decision are: 1) the proportion of non-rejected (usable) data points; and 2) the quantity of data needed to evaluate whether the data is acceptable against the performance standard.

The data will be evaluated for completeness, general conformance with requirements of this QAPP Addendum, and consistency among data sets and with historical data, as appropriate.

Step 4: Defining the Site Boundaries
This Mill Properties includes the Refuse Area and the Oxbow Area (Figure 1).

Step 5: Developing a Decision Rule
The decision on whether data can be used in the TCRA against the performance standard will be based on the validation results. Following validation, the data will be flagged, as appropriate, and any use restrictions noted. The sampling plan has been devised so that the loss of any single data point will not hinder description of the distribution of PCB. Given this, a reasonable decision rule would be that 90% of the data points not be rejected and deemed unusable for evaluation purposes.

Step 6: Limits on Decision Errors
Specifications for this step call for: 1) giving forethought to corrective actions to improve data usability; and 2) understanding the representative nature of the sampling design. This QAPP Addendum has been designed to meet both specifications for this step. The sampling and analysis program has been developed based on a review of previous site data and knowledge of present site conditions. The representative nature of the sampling design has been assured by discussions among professionals familiar with the site and the appropriate government agencies.

Step 7: Design Optimization
The overall QA objective is to develop and implement procedures for field sampling, laboratory analysis, and reporting that will provide data results consistent with the National Contingency Plan (NCP) requirements. Specific procedures for sampling, laboratory instrument calibration, laboratory analysis, data reporting, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP Addendum.

The sampling plan involves a phased approach to both sampling and analysis. This provides the opportunity to evaluate and focus each data collection step to optimize the overall data collection process.

A DQO summary for the sampling TCRA efforts is presented in the subsequent section. The summary consists of stated DQOs relative to data uses, data types, data quantity, sampling and analytical methods, and data measurement performance criteria.

1.4 Data Categories

Three data categories have been defined to address various analytical data uses and the associated QA/QC effort and methods required to achieve the desired levels of quality. These categories are:

Screening Data: Screening data afford a quick assessment of site characteristics or conditions. This DQO is applicable to data collection activities that involve rapid, non-rigorous methods of analysis and QA. This objective is generally applied to physical and/or chemical properties of samples, the degree of contamination relative to concentration differences, and preliminary health and safety assessment.

Screening Data with Definitive Confirmation: Screening data allow rapid identification and quantitation, although the quantitation can be relatively imprecise. This DQO is available for data collection activities that require qualitative and/or quantitative verification of a select portion of sample findings (10% or more). This objective can also be used to verify less rigorous laboratory-based methods.

Definitive Data: Definitive data are generated using analytical methods such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files.

It is anticipated that both screening and definitive data categories will be used during the investigation. Field measurements (e.g., turbidity, ambient air monitoring, dust monitoring, and Photoionization Detector (PID) measurements) that will be obtained during soil sampling for use in qualitatively interpreting other site data will be determined using screening techniques. All remaining parameter measurements will be determined using definitive techniques.

For this project, three levels of data reporting have been defined. They are as follows:

Level 1 - Minimal Reporting: Minimal or "results only" reporting is used for analyses that, due either to their nature (i.e., field monitoring) or the intended data use (i.e., preliminary screening), do not generate or require extensive supporting documentation.

Level 2 - Modified Reporting: Modified reporting is used for analyses that are performed following standard USEPA-approved methods and QA/QC protocols. Based on the intended data use, modified reporting may require some supporting documentation, but not full Contract Laboratory Program- (CLP-) type reporting.

Level 3 - Full Reporting: Full CLP-type reporting is used for those analyses that, based on the intended data use, require full documentation.

The analytical analysis will be performed by Severn Trent Laboratories, Inc. (STL). The analytical results will be reported by the laboratory in the electronic data deliverable (EDD) format presented in Table 1-2. The level analyses reporting requirements are presented in Tables 1-3A thru C.

1.5 Quality Assurance Objectives

The primary QA objective is to develop and implement procedures for defensible sampling, chain-of-custody, laboratory and field analyses, instrument calibration, data reduction and reporting, internal QC, audits, preventative maintenance, and corrective action. These procedures are presented in the 1993 QAPP and in Section 2 of this QAPP Addendum.

1.6 Analytical Laboratory Quality Control Checks

The overarching Analytical Laboratory Quality Control Checks for the project are to implement procedures for defensible sampling, chain-of-custody, laboratory and field analyses, instrument calibration, data reduction and reporting, internal QC, audits, preventive maintenance, and corrective action consistent with the 1993 QAPP.

Specific chemical constituents to be measured, methods to be used for their analysis, and target laboratory reporting limits are listed in Tables 1-1A thru C. Standard operating procedures (SOPs) of laboratory sample preparation and analytical procedure for each method are listed in Table 1-4 and included as Appendix B. The QA/QC sample frequencies and QC measures for laboratory analyses are discussed below and summarized in Tables 1-5 and 1-6.

Laboratory duplicates (splits), laboratory blanks, standards, matrix spikes (MS), matrix spike duplicates (MSD), matrix spike blanks (MSB), field duplicates, trip blanks, and rinse blanks will be analyzed to provide the means for assessing data quality from both the laboratory and field. A brief explanation of each QC sample type is provided below:

- Laboratory duplicates will be used to measure analytical precision.
- Laboratory blanks will be used to assess reagent quality and background from analytical instruments.
- Reference standards/materials will be used to assess analytical accuracy.

- For PCB analyses, MSB analyses will be included to assess method performance in the absence of matrix interference.

- Field duplicates will be used to assess the overall precision of environmental sampling and laboratory analysis.

- Trip blanks will be used for VOC analyses to measure the effects of storage, field sampling, and transport on the samples.

- Rinse blanks will be used to determine the effectiveness of equipment cleaning procedures.

2. Sampling Process and Handling

This section describes how data will be collected, measured, and documented to verify that they will be scientifically sound, of known and documented quality, and suitable for their intended use.

2.1 Sampling Process Design

2.1.1 Post-Excavation Verification Sampling and Analysis

Verification sampling will be conducted on the floor and walls of the Refuse Area and Oxbow Area excavations to confirm that residual PCB concentrations in the remaining soil are at or below the Performance Standard of 10 mg/kg, with a goal of 1 mg/kg. Twenty percent of the verification samples will also be analyzed for target compound list /target analyte list (TCL/TAL) constituents to characterize post-removal soil conditions.

Verification sampling frequency and sampling locations will be determined based on the steps described in *Sampling Strategies and Statistics Training Materials for Part 201 Cleanup Criteria* (MDEQ, 2002). It is anticipated that the sampling grid will be determined in the field for each excavation segment, and samples will be collected in consultation with the USEPA on-scene coordinator following excavation of visible residuals. However, based on the anticipated extent of excavation, proposed sampling grids have been developed as a reference. The anticipated sampling grids and associated calculations are included in Appendix C.

If the analytical results of post-excavation verification samples indicate that PCB are present in soil at concentrations greater than the performance standard, a 20-foot by 20-foot area around the sample location will be re-excavated. A verification sample will then be collected from the floor or wall of the new excavation area and compared to the performance standard. This process will be repeated as necessary to achieve the 10 mg/kg performance standard.

2.1.2 Environmental Monitoring

Environmental monitoring will be conducted throughout the removal action construction activities. Environmental monitoring activities, described below, are anticipated to include dust monitoring, ambient air and water monitoring for PCB, and turbidity monitoring. Additional information regarding environmental monitoring activities is described below.

Dust monitoring will be conducted periodically (i.e., at a minimum of every two hours) by walking the perimeter of active areas during removal action construction activities that may potentially generate dust. Monitoring will consist of both visible observations of airborne particulates as well as monitoring via a Mini-Ram particulate monitor along the perimeter of active areas. In accordance with National Ambient Air Quality Standards (NAAQS), if airborne particulate concentrations are measured at 150 micrograms per cubic meter ($\mu g/m^3$) or above, appropriate dust suppression/control measures will be implemented.

PCB will be monitored in ambient air at two locations (Figure 1), with an action level set at 0.02 $\mu g/m^3$. However, in accordance with the State of Michigan Natural Resources and Environmental Protection Act 451, Rule 225 (3) of Part 55, as amended, a 10-fold increase in the secondary risk screening levels is permitted if the ambient impact occurs on industrial property or public roadways. Given the nature of the physical settings of the removal activities, an action level of 0.2 μg/m^3 for the third location shown on Figure 1 will be used, which will be positioned near the work area. If an action level is exceeded, the USEPA will be notified and corrective actions will be taken to reduce emissions. It should be noted, as conditions change or removal activities move to new locations the air samplers may move to new locations, as well. Any new air sampler location will be selected after consultation with USEPA On Scene Coordinator.

The air monitoring program will follow the procedures outlined by USEPA Method TO-4A from the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air* (USEPA, 1999) for sample collection and analysis. Sampling will be conducted daily for 5 days during commencement of remediation activities at the Mill Properties. Samples will be collected during the entire work day. If the first week's data demonstrate that concentrations at the monitoring locations are below the action levels and similar activities are planned for subsequent weeks, the frequency of sampling may be reduced or terminated upon approval by the USEPA. Following a reduction in sampling frequency, if the nature of the work changes significantly, air monitoring may be resumed.

Meteorological data will be recorded during sampling days. Approximate wind direction, wind speed, and general weather conditions will be obtained from the Battle Creek/Kalamazoo International Airport.

Turbidity monitoring will be performed in the Kalamazoo River approximately 100 feet upstream and 100 feet downstream of excavation activities in the Refuse Area during periods of active work. Measurements of turbidity at the mid-depth point of the water column will be recorded daily (2 hours into the start of the work day). Turbidity monitoring will be conducted consistent with the Remedial Action Turbidity Monitoring Plan (TMP) (BBL, 1999).

If excavation activities progress to within close proximity of the Oxbow Area channel, turbidity monitoring may also be performed at appropriate upstream and downstream locations in the oxbow channel, if necessary.

Water collected from temporary staging/dewatering areas, decontamination fluids, and other liquids generated during construction activities will be treated onsite at a temporary water treatment system (TWTS) located on the South side of the Area East of Davis Creek (Figure 1). The TWTS will consist of filtration and liquid-phase granular two-stage activated carbon. The two-stage activated carbon treatment system will be used so that rotation and replacement of the carbon tanks will occur immediately upon detection of PCB at the intermediate stage. Water will be collected, handled, treated, monitored, and discharged to Davis Creek. To monitor the TWTS, an influent, intermediate (i.e., between the carbon stages), and effluent wastewater sample will be collected and analyzed for PCBs and total suspended solids (TSS) from the TWTS prior to any discharge of the treated water. Treated wastewater will be stored in 20,000 gallon frac tanks until sampling and analysis confirm that the discharge limitations (i.e., 2.6×10^{-5} μg/L for PCBs [or not detected] and 45 mg/L for TSS) have been achieved prior to discharging the water to Davis Creek. Sampling procedures, preservation and handling, and analytical protocol for monitoring for PCB will be consistent with USEPA Method 608 (the quantification level will not exceed 0.1 μg/L). Analytical methods and detection limits used to analyze the water collected during construction activities will be performed consistent with this QAPP Addendum.

2.2 Sampling Procedures and Requirements

Field Standard Operating Procedures (SOPs) for this project are included in the 1993 QAPP and Appendix D of this QAPP Addendum. The SOPs document the procedures that are designed to achieve consistency and comparability in sampling and field measurement techniques implemented by the members of the field sampling team.

The following SOPs will be used for this project:

- Handling, Packing and Shipping Procedures – Appendix A of the 1993 QAPP
- Soil/Residuals Sampling Procedures – Appendix B of the 1993 QAPP;
- Equipment Cleaning Procedures – Appendix C of the 1993 QAPP;
- Photoionization Detector (PID) – Appendix L of the 1993 QAPP;
- Ambient Air Sampling Procedure – Appendix M of the 1993 QAPP;
- Surface Water Sampling Procedures – Appendix N of the 1993 QAPP;
- Turbidity Measurements in Surface Water – *Remedial Action Turbidity Monitoring Plan* (BBL, 1999) and Appendix O of the 1993 QAPP; and
- Dust Monitoring – Appendix D of this QAPP Addendum.

Performance and system audits will be completed during this project to verify that quality data are obtained. These audits are described in Section 10.0 of the 1993 QAPP.

2.3 Sample Handling, Tracking, and Custody Requirements

Sample handling, identification, and chain-of-custody procedures are described in Section 5 and Appendix A of the 1993 QAPP (BBEPC, 1993c). Required containers, preservation techniques, and holding times are provided in Table 2-1.

2.4 Field Analytical Method Requirements

During TCRA construction activities, dust and air monitoring will be conducted with a particulate monitor (MIE MiniRAM, MIE PDR-1200, or equivalent) and PID monitor, respectively, and operated in accordance with the manufacturer's instructions. Turbidity monitoring will be performed in the Kalamazoo River approximately 100 feet upstream and 100 feet downstream of excavation activities in the Refuse Area during periods of active work in accordance with the *Remedial Action Turbidity Monitoring Plan* (BBL, 1999). No other field analysis procedures are required for the TCRA.

In order to maintain field precision and accuracy, all monitors and meters will be calibrated to known standards. Requirements regarding the frequency of required calibrations for field instruments and preventative maintenance are provided in Table 2-2.

2.5 Laboratory Analytical Method Requirements

The maintenance and calibration requirements for the standard fixed laboratory instruments used to perform these analyses are specified in the laboratory-specific SOPs are listed in Table 1-4 and included as Appendix B.

2.5.1 Laboratory Information

Laboratory QA Plans are maintained at the laboratory facilities. See Section 1.1 for laboratory key project personnel and contact information.

2.6 Quality Control Requirements

2.6.1 Field Sampling and Analytical Quality Control

Field sampling QC requirements are summarized in Tables 1-5 and 2-1 which defines the collection frequency and acceptance criteria for the following field QC samples:

- field equipment rinseate blanks;
- field duplicates; and
- sample preservation requirements.

2.6.2 Laboratory Analytical Quality Control

Laboratory analytical QC requirements are described in detail in the published methods (e.g., SW-846). Laboratory SOPs and project-specific requirements are documented in Tables 1-1A thru C and 1-4. If a difference is noted in QC specifications included in the USEPA methods, laboratory SOPs, or Tables 1-1 thru 1-8 of the 1993 QAPP, the Analytical Laboratory Quality Control Checks specified in this 2006 QAPP Addendum take precedence and will be used to evaluate the validity and usability of the data generated during the verification sampling.

Performance and system audits will be completed during this project to maintain high quality data. These audits are described in Section 10.2 of the 1993 QAPP. Preventative maintenance procedures are described in Section 11.2 of the 1993 QAPP.

2.7 Data Reduction, Validation, and Reporting

Data reduction, validation, and reporting procedures will be consistent with Section 8 of the 1993 QAPP, with the exception of the following data validation guidance referenced below:

Analysis	Guidance Documents
Organics	*National Functional Guidelines for Organic Data Review* (USEPA, 1999b).
Inorganics	*National Functional Guidelines for Inorganic Data Review* (USEPA 2002).

Data quality indicators are discussed in Section 12 of the 1993 QAPP.

2.8 Corrective Action

Corrective Action procedures are followed to maintain data quality. Corrective actions are discussed in Section 13 of the 1993 QAPP.

3. References

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Tables

TABLE 1-1A
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS FOR SOIL REMOVAL AND WATER DISCHARGE

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

	Performance Standard		Water (µg/L)		Soil (mg/kg)	
Analyte	Water (µg/L)	Soil (mg/kg)	Laboratory MDL	Laboratory RL	Laboratory MDL	Laboratory RL[1]
PCB (8082)[3,7] - modified						
Aroclor 1016	--	--	0.051	0.05	0.0023	0.017
Aroclor 1221	--	--	0.13	0.05	0.0031	0.017
Aroclor 1232	--	--	0.084	0.05	0.0018	0.017
Aroclor 1242	--	--	0.053	0.05	0.0019	0.017
Aroclor 1248	--	--	0.075	0.05	0.0034	0.017
Aroclor 1254	--	--	0.095	0.05	0.0024	0.017
Aroclor 1260	--	--	0.038	0.05	0.0028	0.017
Total PCB	0.00002	10	0.13	0.05	0.0034	0.017
Volatile Organic Compounds 8260[3]						
Dichlorodifluoromethane	--	--	NC	NC	1.9	10
Chloromethane	--	--	NC	NC	2.5	10
Bromomethane	--	--	NC	NC	2.4	10
Vinyl chloride	--	--	NC	NC	2.6	10
Chloroethane	--	--	NC	NC	2.7	10
Trichlorofluoromethane	--	--	NC	NC	2.7	10
Methylene chloride	--	--	NC	NC	4.5	10
1,1,2-Trichloro-1,2,2-trifluoroethane	--	--	NC	NC	TBD	10
Acetone	--	--	NC	NC	12	30
Carbon disulfide	--	--	NC	NC	2.3	10
Methyl acetate	--	--	NC	NC	TBD	10
1,1-Dichloroethene	--	--	NC	NC	1.7	10
1,1-Dichloroethane	--	--	NC	NC	1.6	10
trans-1,2-Dichloroethene	--	--	NC	NC	1.9	10
Methyl tert-butyl ether	--	--	NC	NC	1.4	10
Chloroform	--	--	NC	NC	1.6	10
1,2-Dichloroethane	--	--	NC	NC	1.2	10
cis-1,2-Dichloroethene	--	--	NC	NC	1.8	10
2-Butanone	--	--	NC	NC	1.7	10
1,1,1-Trichloroethane	--	--	NC	NC	1.6	10
Cyclohexane	--	--	NC	NC	TBD	10
Carbon tetrachloride	--	--	NC	NC	1.4	10
Bromodichloromethane	--	--	NC	NC	1.3	10
1,2-Dichloropropane	--	--	NC	NC	1.4	10
cis-1,3-Dichloropropene	--	--	NC	NC	1.3	10
Trichloroethene	--	--	NC	NC	1.7	10
Methylcyclohexane	--	--	NC	NC	TBD	10
Dibromochloromethane	--	--	NC	NC	1.2	10
1,2-Dibromoethane	--	--	NC	NC	1.8	10
1,1,2-Trichloroethane	--	--	NC	NC	1.5	10
Benzene	--	--	NC	NC	1.6	10
trans-1,3-Dichloropropene	--	--	NC	NC	1.6	10
Bromoform	--	--	NC	NC	0.56	10
Isopropylbenzene	--	--	NC	NC	2.0	10
4-Methyl-2-pentanone	--	--	NC	NC	4.1	10
2-Hexanone	--	--	NC	NC	7.4	10
Tetrachloroethene	--	--	NC	NC	1.7	10
Toluene	--	--	NC	NC	1.5	10
1,1,2,2-Tetrachloroethane	--	--	NC	NC	1.6	10
Chlorobenzene	--	--	NC	NC	1.5	10
Ethylbenzene	--	--	NC	NC	1.5	10
Styrene	--	--	NC	NC	9.2	10
Xylenes (total)	--	--	NC	NC	8.3	10
1,3-Dichlorobenzene	--	--	NC	NC	1.2	10
1,4-Dichlorobenzene	--	--	NC	NC	1.3	10
1,2-Dichlorobenzene	--	--	NC	NC	1.2	10
1,2-Dibromo-3-chloropropane	--	--	NC	NC	1.7	10
1,2,4-Trichlorobenzene	--	--	NC	NC	1.3	10

See Notes on Page 3.

TABLE 1-1A
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS FOR SOIL REMOVAL AND WATER DISCHARGE

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

	Performance Standard		Water (µg/L)		Soil (mg/kg)	
Analyte	Water (µg/L)	Soil (mg/kg)	Laboratory MDL	Laboratory RL	Laboratory MDL	Laboratory RL[1]
Semivolatile Organic Compounds 8270[3]						
Benzaldehyde	--	--	NC	NC	TBD	330
Phenol	--	--	NC	NC	93	330
bis(2-Chloroisopropyl)ether	--	--	NC	NC	88	330
2-Chlorophenol	--	--	NC	NC	68	330
2-Methylphenol	--	--	NC	NC	76	330
2,2'-oxybis(1-Chloropropane)	--	--	NC	NC	TBD	330
Acetophenone	--	--	NC	NC	TBD	330
4-Methylphenol	--	--	NC	NC	73	330
N-Nitrosos-di-n-propylamine	--	--	NC	NC	100	330
Hexachloroethane	--	--	NC	NC	83	330
Nitrobenzene	--	--	NC	NC	79	330
Isophorone	--	--	NC	NC	70	330
2-Nitrophenol	--	--	NC	NC	78	330
2,4-Dimethylphenol	--	--	NC	NC	71	330
bis(2-Chloroethoxy)methane	--	--	NC	NC	81	330
2,4-Dichlorophenol	--	--	NC	NC	67	330
Naphthalene	--	--	NC	NC	71	330
4-Chloroaniline	--	--	NC	NC	75	330
Hexachlorobutadiene	--	--	NC	NC	75	330
Caprolactam	--	--	NC	NC	TBD	330
4-Chloro-3-methylphenol	--	--	NC	NC	39	330
2-Methylnaphthalene	--	--	NC	NC	63	330
Hexachlorocyclopentadiene	--	--	NC	NC	78	330
2,4,6-Trichlorophenol	--	--	NC	NC	45	330
2,4,5-Trichlorophenol	--	--	NC	NC	45	830
1,1'-Biphenyl	--	--	NC	NC	TBD	330
2-Chloronaphthalene	--	--	NC	NC	64	330
2-Nitroaniline	--	--	NC	NC	43	830
Dimethylphthalate	--	--	NC	NC	46	330
Acenaphthylene	--	--	NC	NC	49	330
2,6-Dinitrotoluene	--	--	NC	NC	44	330
3-Nitroaniline	--	--	NC	NC	51	830
Acenaphthene	--	--	NC	NC	57	330
2,4-Dinitrophenol	--	--	NC	NC	270	830
4-Nitrophenol	--	--	NC	NC	140	830
Dibenzofuran	--	--	NC	NC	48	330
2,4-Dinitrotoluene	--	--	NC	NC	47	330
Diethylphthalate	--	--	NC	NC	48	330
4-Chlorophenyl-phenylether	--	--	NC	NC	51	330
Fluorene	--	--	NC	NC	52	330
4-Nitroaniline	--	--	NC	NC	72	830
4,6-Dinitro-2-methylphenol	--	--	NC	NC	210	830
N-Nitrosodiphenylamine	--	--	NC	NC	68	330
4-Bromophenyl-phenylether	--	--	NC	NC	61	330
Hexachlorobenzene	--	--	NC	NC	59	330
Atrazine	--	--	NC	NC	TBD	330
Pentachlorophenol	--	--	NC	NC	180	830
Phenanthrene	--	--	NC	NC	47	330
Anthracene	--	--	NC	NC	49	330
Carbazole	--	--	NC	NC	56	330
Di-n-butyl phthalate	--	--	NC	NC	61	330
Fluoranthene	--	--	NC	NC	56	330
Pyrene	--	--	NC	NC	36	330
Butylbenzylphthalate	--	--	NC	NC	76	330
3,3'-Dichlorobenzidine	--	--	NC	NC	380	330
Benzo(a)anthracene	--	--	NC	NC	52	330
Chrysene	--	--	NC	NC	44	330
bis(2-Ethylhexyl)phthalate	--	--	NC	NC	74	330
Di-n-octyl phthalate	--	--	NC	NC	61	330
Benzo(b)fluoranthene	--	--	NC	NC	38	330
Benzo(k)fluoranthene	--	--	NC	NC	31	330
Benzo(a)pyrene	--	--	NC	NC	45	330
Indeno(1,2,3-cd)pyrene	--	--	NC	NC	55	330
Dibenzo(a,h)anthracene	--	--	NC	NC	66	330
Benzo(g,h,i)perylene	--	--	NC	NC	66	330

See Notes on Page 3.

TABLE 1-1A
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS FOR SOIL REMOVAL AND WATER DISCHARGE

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

	Performance Standard		Water (µg/L)		Soil (mg/kg)	
Analyte	Water (µg/L)	Soil (mg/kg)	Laboratory MDL	Laboratory RL	Laboratory MDL	Laboratory RL[1]
Inorganics 6010[3]						
Aluminum	--	--	NC	NC	2.2	40
Antimony	--	--	NC	NC	3.7	12
Arsenic	--	--	NC	NC	0.54	3
Barium	--	--	NC	NC	TBD	40
Beryllium	--	--	NC	NC	0.04	1
Cadmium	--	--	NC	NC	0.063	1
Calcium	--	--	NC	NC	4.4	1000
Chromium	--	--	NC	NC	0.14	2
Cobalt	--	--	NC	NC	0.11	10
Copper	--	--	NC	NC	0.24	5
Iron	--	--	NC	NC	3.1	20
Lead	--	--	NC	NC	0.31	2
Magnesium	--	--	NC	NC	0.8	1000
Manganese	--	--	NC	NC	0.21	3
Nickel	--	--	NC	NC	0.12	8
Potassium	--	--	NC	NC	8.4	1000
Selenium	--	--	NC	NC	1.1	7
Silver	--	--	NC	NC	0.15	2
Sodium	--	--	NC	NC	42	1000
Thallium	--	--	NC	NC	2.4	5
Vanadium	--	--	NC	NC	0.095	10
Zinc	--	--	NC	NC	0.64	12
Inorganics 7471[3]						
Mercury	--	--	NC	NC	0.014	0.1
Waste Management						
Reactive Cyanide (9012)[3]	--	--	NC	NC	TBD	TBD
Reactive Sulfide (9380/9034)[3]	--	--	NC	NC	TBD	TBD
Ignitability (1010)[3]	--	--	NC	NC	TBD	TBD
Corrosivity (pH) (9045)[3]	--	--	NC	NC	TBD	TBD
Paint Filter Liquids (9095)[3]	--	--	NC	NC	TBD	TBD
Total Suspended Solids (160.2)[4]	45000	--	--	4000	TBD	TBD
Backfill Soils						
Corrosivity (pH) (9045)[4]	--	--	NC	NC	TBD	TBD
TOC (Lloyd Kahn)[5]	--	--	NC	NC		

Notes:
1. The target reporting limits are based on wet weight. The actual reporting limits will vary based on sample weight and moisture content.
2. See Section 3.7.2 of the Work Plan for additional information.
3. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed.* Washington, DC. 1996.
4. USEPA. *Methods for Chemical Analysis of Water and Wastes*. EPA/600/4-79/020. EMSL-Cincinnati. 1983.
5. USEPA. *Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method).* July 27, 1988.
6. Laboratory will use the most current version of each method, as it is promulgated.
7. Standard Operating Procedure (SOP) modified by Blasland, Bouck & Lee (BBL)/Severn Trent Laboratories, Inc. (STL) consistent with USEPA SW-846 8082 Method.

NC = Not Collected
-- = No performance Standard required, analyses performed for post-removal characterization
TBD = To Be Determined.
RL = Reporting Limit
MDL = Method Detection Limit
mg/kg = Milligrams per kilogram
µg/L = Micrograms per liter
TOC = Total Organic Carbon
PCB = Polychlorinated Biphenyls

TABLE 1-1B
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS FOR AMBIENT AIR AND DUST

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Analyte	Action Level[2] ($\mu g/m^3$)	Air (µg/PUF)	
		Laboratory MDL	Laboratory RL
PCB (TO-4A[1])			
Aroclor 1016	--	0.013	0.10
Aroclor 1221	--	0.042	0.10
Aroclor 1232	--	0.03	0.10
Aroclor 1242	--	0.034	0.10
Aroclor 1248	--	0.055	0.17
Aroclor 1254	--	0.029	0.13
Aroclor 1260	--	0.0095	0.10
Total PCB	0.02/0.2	0.055	0.10
Dust	150	NA	NA

Notes:
1. USEPA. *Compendium Method TO-4A: Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)*. EPA/625/R-96/010b. 1999.
2. Ambient Air action levels are based on the location of monitors described in the section 3.7.2 of the TCRA Work Plan (Work Plan) (BBL, 2006)
PCB - Polychlorinated Biphenyl
MDL = Method Detection Limit
RL = Reporting Limit
µg/m3 = Micrograms per meters cubed
µg/PUF = Micrograms per PUF
NA - Not Applicable

TABLE 1-1C
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS FOR DISPOSAL

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Analyte	Regulatory Level[3] (mg/L)	Leachates (mg/L) Laboratory MDL	Leachates (mg/L) Laboratory RL
TCLP-Volatiles 1311/8260			
Benzene	0.5	0.00013	0.005
Chlorobenzene	100	0.00013	0.005
Carbon tetrachloride	0.5	0.00019	0.005
Chloroform	6	0.00017	0.005
1,2-Dichloroethane	0.5	0.00016	0.005
1,1-Dichloroethene	0.7	0.00014	0.005
2-Butanone	200	0.00042	0.005
Tetrachloroethene	0.7	0.0005	0.005
Trichloroethene	0.5	0.00024	0.005
Vinyl chloride	0.2	0.00017	0.005
TCLP-Semivolatiles 1311/8270			
2-Methylphenol	200	0.0011	0.05
2. Ambient Air action levels are based on the loca	7.5	0.0017	0.05
2,4-Dinitrotoluene	0.13	0.0018	0.05
Hexachlorobenzene	0.13	0.0013	0.05
Hexachlorobutadiene	0.5	0.0019	0.05
Hexachloroethane	3	0.002	0.05
Nitrobenzene	2	0.0013	0.05
NA - Not Applicable	100	0.0021	0.13
Pyridine	5	0.002	0.05
2,4,5-Trichlorophenol	400	0.0014	0.13
2,4,6-Trichlorophenol	2	0.0011	0.06
TCLP-Metals 1311/6010			
Arsenic	5	0.0051	0.01
Barium	100	0.0062	0.2
Cadmium	1	0.00078	0.005
Chromium	5	0.0023	0.001
Lead	5	0.0017	0.001
Selenium	1	0.005	0.0035
Silver	5	0.0026	0.001
TCLP-Metals1311/7470			
Mercury	0.2	0.0000991	0.0002

Notes:
1. See Section 3.7.2 of the Work Plan for additional information.
2. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed.* Washington, DC. 1996.
3. TCLP regulatory level limits are based on USEPA 40 CFR Part 261
MDL = Method Detection Limit
RL = Reporting limit
TCLP = Toxicity Characterization Leaching Procedure
mg/L = milligrams per liter

TABLE 1-2
ELECTRONIC DATA DELIVERABLE FORMAT

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Field Name	Maximum Length	Data Type	Comments
FIELD SAMPLE ID	50	TEXT	From the chain of custody. Add "RE" or "DL" to differentiate reanalyses and dilutions.
SDG	50	TEXT	
LAB SAMPLE ID	50	TEXT	
MATRIX	10	TEXT	SOIL, WATER, SEDIMENT, etc.
SAMPLE TYPE	10	TEXT	FB, RB, TB, FD, FS for Field Blank, Rinse Blank, Trip Blank, Field Duplicate and Field Sample, respectively. DEFAULT TO FS
DATE COLLECTED	--	DATE/TIME	MM/DD/YY
TIME COLLECTED*	--	DATE/TIME	Military time
DEPTH START	--	NUMBER	
DEPTH END	--	NUMBER	
DEPTH UNITS	25	TEXT	FEET, INCHES METERS, etc.
ANALYTICAL METHOD	50	TEXT	
CAS NUMBER	25	TEXT	
ANALYTE	100	TEXT	
2. Ambient Air action level	10	TEXT	"U" for non-detected, others as defined by laboratory.
REPORTING LIMIT	--	NUMBER	
RESULT UNIT	25	TEXT	
DILUTION FACTOR	--	NUMBER	
REPORTABLE RESULT	--	YES/NO	DEFAULT TO YES
FILTERED?	--	YES/NO	
NA - Not Applicable	--	DATE/TIME	MM/DD/YY
TIME ANALYZED*	--	DATE/TIME	Military time
DATE EXTRACTED*	--	DATE/TIME	MM/DD/YY
LABORATORY NAME*	50	TEXT	

Notes:
1 This definition is for an "Excel-type" spreadsheet. Fields flagged with an "*" are optional and may be left blank if not available electronically from the laboratory.
2 Depth-related fields may be left blank for samples and matrices for which they are not applicable.
SDG = Sample Delivery Group

TABLE 1-3A
WATER LABORATORY REPORTING REQUIREMENTS

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Data Type	Data Quality Level[1]
TARGET CONSTITUENTS	
Total PCB	III
Total Suspended Solids	II

Notes:
1. Data Quality Levels:
   - III Analysis using USEPA SW-846 methods, with CLP-type documentation
   - II Laboratory analysis using methods other than CLP, without CLP documentation

PCB = Polychlorinated Biphenyls
CLP = Contract Laboratory Program

TABLE 1-3B
SOIL LABORATORY REPORTING REQUIREMENTS

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Data Type	Data Quality Level[1]
SOIL TARGET CONSTITUENTS	
PCB	III
TCL VOCs, SVOCs	III
TAL Metals	III
TCLP VOCs, SVOCs, and Metals; RCRA Reactivity; Ignitability; and Corrosivity	II
Paint Filter Liquids	I
BACKFILL TARGET CONSTITUENTS	
pH	II
TOC	II

Notes:

1. Data Quality Levels:
   - III Analysis using USEPA SW-846 methods, with CLP-type documentation
   - II Laboratory analysis using methods other than CLP, without CLP documentation.
   - I Field Screening

PCB = Polychlorinated Biphenyls
TCL = Target Compound List
VOCs = Volatile Organic Compounds
SVOCs = Semivolatile Organic Compounds
RCRA = Resource Conservation and Recovery Act
TOC = Total Organic Carbon
CLP = Contract Laboratory Program
TCLP = Toxicity Characterization Leaching Potential
TAL = Target Analyte List

TABLE 1-3C
AIR LABORATORY REPORTING REQUIREMENTS

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Data Type	Data Quality Level[1]
PCB	III
Airborne Particulates	I

Notes:
1. Data Quality Levels:
   II Analysis using TO-4A methods, with CLP-type documentation
   I Field screening

PCB = Polychlorinated Biphenyls
CLP = Contract Laboratory Program

TABLE 1-4
LABORATORY STANDARD OPERATING PROCEDURES

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION - KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Appendix	Method Number	Title
B-1	SW-846 8082	Polychlorinated Biphenyls (PCB) by Gas Chromatography/Electron Capture Detectore (GC/ECD)
B-2	SW-846 7471A	Mercury (Cold Vapor Technique)
B-3	SW-846 6010B	Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)
B-4	SW-846 3010	Acid Digestion of Waters for Total Metals
B-5	SW-846 3050	Acid Digestion of Soils, Sediments, & Sludge for Total Metals ICP-AES and ICP-Mass Spectrometer (MS)
B-6	SW-846 8270C	Semivolatile Organic Compounds by GC/MS
B-7	SW-846 5035	Closed-System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples
B-8	SW-846 8260B	Volatile Organic Compounds by (GC/MS)
B-9	SW-846 1311/Non-Volatile TCLP	Toxicity Characteristic Leaching Procedure (TCLP)
B-10	SW-846 3510C	Separatory Funnel Liquid-Liquid Extraction
B-11	SW-846 3550B	Ultrasonic Extraction
B-12	Cleanup	Extract Cleanup: Florisil, Silica Gel, Sulfur, Sulfuric Acid
B-13	SW-846 3640	Gel Permeation Cleanup
B-14	WC-1010	Ignitability by Pensky-Martens Closed-Cup Tester
B-15	WC-160.2	The Determination of Non-Filterable Residue (Total Suspended Solids - TSS)
B-16	SW-846 9030B & SW-846 9034	Acid-Soluble Sulfide: Distillation and Titration
B-17	EPA 335.2, 335.3, 335.4	Total and Amenable Cyanide
B-18	WC-Percent Solids	Percent Solids Determination
B-19	EPA 150.1	Determination of pH
B-20	Lloyd Kahn	Total Organic Carbon (TOC) in Soils and Sediment
B-21	TO-4A	Extraction of Toxic Organic Compounds in Ambient Air
B-22	SW-846 3540C	Soxhlet Extraction
B-23	SW-846 9095A	Paint Filter Liquids Test

TABLE 1-5
SAMPLE QUANTITIES AND QUALITY CONTROL FREQUENCIES

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Parameter	Estimated Environmental Sample Quality	Field QC Analyses						Laboratory QC Sample						Total
		Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
		Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Soil														
PCB	67	NA	--	1/10	6	1/10	6	1/20	3	1/20	3	NA	--	85
Reactivity	2	NA	--	NA	--	1/20	1	1/20	--	NA	--	1/20	1	4
Corrosivity	2	NA	--	NA	--	1/20	1	NA	--	NA	--	1/20	1	4
Ignitability	2	NA	--	NA	--	1/20	1	NA	--	NA	--	1/20	1	4
TCLP-Volatiles	2	NA	--	NA	--	1/20	1	1/20	1	NA	--	NA	--	4
TCLP-Semivolatiles	2	NA	--	NA	--	1/20	1	1/20	1	NA	--	NA	--	4
TCLP-Metals	2	NA	--	NA	--	1/20	1	1/20	1	NA	--	NA	--	4
Paint Filter Liquids	TBD	NA	--	NA	--	1/20	--	NA	--	NA	--	1/20	--	TBD
Volatile Organic Compounds	11	1/cooler		1/10	2	1/10	2	1/20	1	1/20	1	NA	--	17
Semivolatile Organic Compounds	11	NA	--	1/10	2	1/10	2	1/20	1	1/20	1	NA	--	17
Metals	11	NA	--	1/10	2	1/10	2	1/20	1	NA	--	1/20	1	17
2. Ambient Air action levels are based on the loca	TBD	NA	--	1/10	--	1/10	--	NA	--	NA	--	1/20	--	TBD
Total Organic Carbon	TBD	NA	--	1/10	--	1/10	--	1/20	--	NA	--	1/20	--	TBD
Air														
PCB	TBD	1/day	TBD	NA	--	1/day	TBD	NA	--	NA	--	NA	--	TBD
Water														
PCB	TBD	NA	--	NA	--	NA	--	1/20	--	1/20	--	NA	--	TBD
NA - Not Applicable	TBD	NA	--	NA	--	1/10	--	NA	--	NA	--	1/20	--	TBD

Notes:
Sample counts are an approximation.
Field duplicates will consist of co-located samples for air analysis.
1/day = One rinse blank per day or one per 20 samples, whichever is more frequent. Rinse blanks not required when dedicated sampling equipment is used.
Freq = Frequency
NA = Not Applicable
No. = Number
PCB = Polychlorinated Biphenyls
QC = Quality Control
TBD = To Be Determined
TCLP = Toxicity Characteristic Leaching Procedure

TABLE 1-6
ANALYTICAL QUALITY CONTROL LIMITS[1]

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Parameter	Accuracy - % Recovery			Precision - RPD		
	Surrogate	**MS/MSD**	**LCS**	**MS/MSD**	**Lab Duplicate**	**Field Duplicate**
Soil						
PCB	60-150	29-150	29-150	30	--	100
Volatile Organic Compounds	60-140	60-140	70-140	25	--	100
Semivolatile Organic Compounds	20-140	20-140	40-120	40	--	100
Metals	--	80-120	80-120	--	20	100
Backfill Soil						
2. Ambient Air action levels are based	--	75-125	75-125	--	30	100
Air						
PCB	60-140	40-130	50-140	20	--	100
Water						
PCB	30-150	40-130	50-140	20	--	50
TSS	--	70-130	70-130	--	30	50

NA - Not Applicable

Note:

1. The listed QC limits are based on SW-846 guidance and are advisory. The actual limits are determined based on laboratory performance. Frequent failure to meet the QC limits, however, warrant investigation of the laboratory.

MS = Matrix Spike
MSD = Matrix Spike Duplicate
LCS = Laboratory Control Sample
RPD = Relative Percent Difference
PCB = Polychlorinated Biphenyls
TSS = Total Suspended Solids

TABLE 2-1
SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Parameter	Method[5]	Bottle Type	Preservation	Holding Time[6]
Soil				
Reactive Cyanide	9012 (3)	One 8-oz wide mouth glass jar	Cool to 4°C	14 days to analysis
Reactive Sulfide	9030/9034 (3)			7 days to analysis
Ignitability	1010 (3)			none established
Corrosivity	9045 (3)			48 hours to analysis
TCLP-Volatiles	1311/8260 (3)	One 4-oz glass jar with Teflon®-lined lid	Cool to 4°C	14 days to TCLP extraction
				14 days to analysis
TCLP-Semivolatiles	1311/6010 (3)	One 8-oz glass jar with Teflon®-lined lid	Cool to 4°C	14 days to TCLP extraction
				7 days to extract prep
				40 days to analysis
TCLP-Metals (except mercury)	1311/6010 (3)		Cool to 4°C	180 days to TCLP extraction
				180 days to analysis
TCLP-Mercury	1311/7470 (3)			28 days to TCLP extraction
				28 days to analysis
Paint Filter Liquids	9095 (3)	One 4-oz wide mouth glass jar	Cool to 4°C±2°C	none established
Volatile Organic Compounds	8260 (3)	3-EnCore™ samplers One 40-ml glass vial	Cool to 4°C±2°C	48 hours to preservation
				14 days to analysis
Semivolatile Organic Compounds	8270 (3)	One 8-oz glass jar with Teflon®-lined lid	Cool to 4°C±2°C	14 days to extraction
				40 days to analysis
PCB	8082 (3)			14 days to extraction
NA - Not Applicable				40 days to analysis
Metals (except mercury)	6010 (3)	One 4-oz wide mouth glass jar	Cool to 4°C±2°C	180 days to analysis
Mercury	7471 (3)			28 days to analysis
Backfill Soil				
pH	9045 (3)	One 4-oz wide mouth glass jar	Cool to 4°C±2°C	ASAP
TOC	Lloyd Kahn (4)		Cool to 4°C	14 days to analysis
Air				
PCB	TO-4A (2)	Polyurethane Foam (PUF) Cartridge	Cool to 4°C	7 days to extract prep
				40 days to analysis
Water				
PCB	8082 (3)	Two 1-L amber glass bottle with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction 40 days to analysis
TSS	160.2 (1)	One 1-L plastic bottle	Cool to 4°C±2°C	7 days to analysis

Notes:
1. USEPA. *Methods for Chemical Analysis of Water and Wastes*. EPA/600/4-79/020. EMSL-Cincinnati. 1983.
2. USEPA. *Compendium Method TO-4A: Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)*. EPA/625/R-96/010b. 1999.
3. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846.* 3rd ed. Washington, DC. 1996.
4. USEPA. *Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method).* July 27, 1988.
5. Laboratory will use the most current version of each method, as it is promulgated.
6. All holding times are measured from date of collection.

°C = Degrees Celsius
mL/L = Milliliters per liter
PCB = Polychlorinated Biphenyls
TOC = Total Organic Carbon
TSS = Total Suspended Solids
TCLP = Toxicity Characteristic Leaching Procedure
ASAP = As Soon As Possible

TABLE 2-2
FIELD EQUIPMENT CALIBRATION REQUIREMENTS AND PREVENTATIVE MAINTENANCE SCHEDULE

QUALITY ASSURANCE PROJECT PLAN ADDENDUM
GEORGIA-PACIFIC CORPORATION – KALAMAZOO MILL PROPERTY AND FORMER HAWTHORNE MILL PROPERTY
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE

Instrument	Task	Frequency
Photoionization Detector (PID)	a) Calibrate b) Inspect c) Check/recharge Batteries d) Store in Protective Case e) Return to manufacturer for service	a) Daily or if/when erroneous readings are suspected b) Daily c) Daily d) After each use e) As needed
Particulate Monitor	a) Calibrate b) Inspect c) Check batteries, replace if necessary d) Store in protective case e) Clean fan and dust inside probe	a) Daily b) Daily c) Daily d) After each use e) Once every 2 weeks of use or as needed
Nephelometer (Turbidity)	a) Store in protective case b) Inspect c) Clean sample cells d) Clean lens e) Check batteries, and recharge if necessary f) Calibrate g) Return to manufacturer for service	a) After each use b) After each use c) Daily d) Daily e) Daily f) Daily g) As needed
High-Volume Polyurethane Foam (PUF) Air Sampler	a) Calibrate b) Inspect c) Clean exterior of sampler d) Clean glassware e) Return to manufacturer for service	a) When new and before each use b) Daily c) Weekly d) Daily e) As needed

Figure

Appendices

Appendix A

Laboratory NELAP Accreditation

State of New Jersey
Department of Environmental Protection

Certifies That

STL Burlington

***Laboratory Certification ID#*: VT972**

having duly met the requirements of the

Regulations Governing The Certification Of
Laboratories And Environmental Measurements N.J.A.C. 7:18 et. seq.

and

having been found compliant with the standards approved by the

National Environmental Laboratory Accreditation Conference

is hereby approved as a

State Certified Environmental Laboratory

to perform the analyses as indicated on the Annual Certified Parameter List
which must accompany this certificate to be valid

Expiration Date June 30, 2006

NJDEP is a NELAP Recognized Accrediting Authority

Joseph F. Aiello, Chief
Office of Quality Assurance

THIS CERTIFICATE IS TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY WITH THE ANNUAL CERTIFIED PARAMETER LIST IN A LOCATION ON THE PREMISES VISIBLE TO THE PUBLIC

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: CAP01 – Atmos. Inorg. Parameters, Non-Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CAP01.00005	AE	Thermal Conductivity	[EPA 3C]	Carbon Dioxide
Certified	Yes	NJ	CAP01.00068	AE	Thermal Conductivity	[EPA 3C]	Methane
Certified	Yes	NJ	CAP01.00070	AE	Thermal Conductivity	[EPA 3C]	Nitrogen
Certified	Yes	NJ	CAP01.00100	AE	Thermal Conductivity	[EPA 3C]	Oxygen

Category: CAP03 – Atmospheric Organic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CAP03.00015	AE	FID	[EPA 25C]	Non-Methane Organic Compounds
Certified	Yes	NJ	CAP03.00184	AE	GC/MS, Canisters	[EPA TO-15]	Acetone
Certified	Yes	NJ	CAP03.00225	AE	GC/MS, Canisters	[EPA TO-15]	Benzene
Certified	Yes	NJ	CAP03.00250	AE	GC/MS, Canisters	[EPA TO-15]	Bromodichloromethane
Certified	Yes	NJ	CAP03.00255	AE	GC/MS, Canisters	[EPA TO-15]	Bromoform
Certified	Yes	NJ	CAP03.00260	AE	GC/MS, Canisters	[EPA TO-15]	Bromomethane
Certified	Yes	NJ	CAP03.00265	AE	GC/MS, Canisters	[EPA TO-15]	Butadiene (1,3-)
Certified	Yes	NJ	CAP03.00270	AE	GC/MS, Canisters	[EPA TO-15]	Carbon disulfide
Certified	Yes	NJ	CAP03.00275	AE	GC/MS, Canisters	[EPA TO-15]	Carbon tetrachloride
Certified	Yes	NJ	CAP03.00300	AE	GC/MS, Canisters	[EPA TO-15]	Chlorobenzene
Certified	Yes	NJ	CAP03.00305	AE	GC/MS, Canisters	[EPA TO-15]	Chloroethane
Certified	Yes	NJ	CAP03.00310	AE	GC/MS, Canisters	[EPA TO-15]	Chloroform
Certified	Yes	NJ	CAP03.00315	AE	GC/MS, Canisters	[EPA TO-15]	Chloromethane
Certified	Yes	NJ	CAP03.00325	AE	GC/MS, Canisters	[EPA TO-15]	Chlorotoluene (2-)
Certified	Yes	NJ	CAP03.00335	AE	GC/MS, Canisters	[EPA TO-15]	Cyclohexane
Certified	Yes	NJ	CAP03.00342	AE	GC/MS, Canisters	[EPA TO-15]	Dibromochloromethane
Certified	Yes	NJ	CAP03.00350	AE	GC/MS, Canisters	[EPA TO-15]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	CAP03.00355	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorobenzene (1,2-)
Certified	Yes	NJ	CAP03.00360	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	CAP03.00365	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	CAP03.00368	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorodifluoromethane
Certified	Yes	NJ	CAP03.00370	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethane (1,1-)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: CAP03 -- Atmospheric Organic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CAP03.00375	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethane (1,2-)
Certified	Yes	NJ	CAP03.00380	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethene (1,1-)
Certified	Yes	NJ	CAP03.00384	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	CAP03.00385	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethene (trans-1,2-)
Certified	Yes	NJ	CAP03.00395	AE	GC/MS, Canisters	[EPA TO-15]	Dichloropropane (1,2-)
Certified	Yes	NJ	CAP03.00400	AE	GC/MS, Canisters	[EPA TO-15]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	CAP03.00401	AE	GC/MS, Canisters	[EPA TO-15]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	CAP03.00405	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorotetrafluoroethane (1,2-)
Certified	Yes	NJ	CAP03.00440	AE	GC/MS, Canisters	[EPA TO-15]	Dioxane (1,4-)
Certified	Yes	NJ	CAP03.00465	AE	GC/MS, Canisters	[EPA TO-15]	Ethylbenzene
Certified	Yes	NJ	CAP03.00480	AE	GC/MS, Canisters	[EPA TO-15]	Ethyltoluene (4-)
Certified	Yes	NJ	CAP03.00490	AE	GC/MS, Canisters	[EPA TO-15]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	CAP03.00498	AE	GC/MS, Canisters	[EPA TO-15]	Hexanone (2-)
Certified	Yes	NJ	CAP03.00500	AE	GC/MS, Canisters	[EPA TO-15]	Heptane (n-)
Certified	Yes	NJ	CAP03.00505	AE	GC/MS, Canisters	[EPA TO-15]	Hexane (n-)
Certified	Yes	NJ	CAP03.00511	AE	GC/MS, Canisters	[EPA TO-15]	Isopropanol
Certified	Yes	NJ	CAP03.00525	AE	GC/MS, Canisters	[EPA TO-15]	Methyl ethyl ketone
Certified	Yes	NJ	CAP03.00535	AE	GC/MS, Canisters	[EPA TO-15]	Methyl isobutyl ketone
Certified	Yes	NJ	CAP03.00550	AE	GC/MS, Canisters	[EPA TO-15]	Methyl tert-butyl ether
Certified	Yes	NJ	CAP03.00555	AE	GC/MS, Canisters	[EPA TO-15]	Methylene chloride (Dichloromethane)
Certified	Yes	NJ	CAP03.00625	AE	GC/MS, Canisters	[EPA TO-15]	Styrene
Certified	Yes	NJ	CAP03.00635	AE	GC/MS, Canisters	[EPA TO-15]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	CAP03.00640	AE	GC/MS, Canisters	[EPA TO-15]	Trimethylbenzene (1,3,5-)
Certified	Yes	NJ	CAP03.00645	AE	GC/MS, Canisters	[EPA TO-15]	Trimethylbenzene (1,2,4-)
Certified	Yes	NJ	CAP03.00650	AE	GC/MS, Canisters	[EPA TO-15]	Trimethylpentane (2,2,4-)
Certified	Yes	NJ	CAP03.00652	AE	GC/MS, Canisters	[EPA TO-15]	Tert-butyl alcohol
Certified	Yes	NJ	CAP03.00655	AE	GC/MS, Canisters	[EPA TO-15]	Tetrachloroethane (1,1,2,2-)
Certified	Yes	NJ	CAP03.00660	AE	GC/MS, Canisters	[EPA TO-15]	Tetrachloroethene
Certified	Yes	NJ	CAP03.00665	AE	GC/MS, Canisters	[EPA TO-15]	Toluene
Certified	Yes	NJ	CAP03.00670	AE	GC/MS, Canisters	[EPA TO-15]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	CAP03.00675	AE	GC/MS, Canisters	[EPA TO-15]	Trichloroethane (1,1,2-)
Certified	Yes	NJ	CAP03.00680	AE	GC/MS, Canisters	[EPA TO-15]	Trichloroethene

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: CAP03 -- Atmospheric Organic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Applied	No	NJ	CAP03.00684	AE	GC/MS, Canisters	[EPA TO-15]	Trichlorofluoromethane
Certified	Yes	NJ	CAP03.00685	AE	GC/MS, Canisters	[EPA TO-15]	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
Certified	Yes	NJ	CAP03.00710	AE	GC/MS, Canisters	[EPA TO-15]	Vinyl chloride
Certified	Yes	NJ	CAP03.00715	AE	GC/MS, Canisters	[EPA TO-15]	Xylene (m-)
Certified	Yes	NJ	CAP03.00720	AE	GC/MS, Canisters	[EPA TO-15]	Xylene (o-)
Certified	Yes	NJ	CAP03.00725	AE	GC/MS, Canisters	[EPA TO-15]	Xylene (p-)
Certified	Yes	NJ	CAP03.00730	AE	GC/MS, Canisters	[EPA TO-15]	Xylenes (total)
Certified	Yes	NJ	CAP03.06150	AE	GC/MS	[EPA TO-13A]	Acenaphthene
Certified	Yes	NJ	CAP03.06160	AE	GC/MS	[EPA TO-13A]	Acenaphthylene
Certified	Yes	NJ	CAP03.06170	AE	GC/MS	[EPA TO-13A]	Anthracene
Certified	Yes	NJ	CAP03.06180	AE	GC/MS	[EPA TO-13A]	Benzo(a)anthracene
Certified	Yes	NJ	CAP03.06190	AE	GC/MS	[EPA TO-13A]	Benzo(a)pyrene
Certified	Yes	NJ	CAP03.06200	AE	GC/MS	[EPA TO-13A]	Benzo(b)fluoranthene
Certified	Yes	NJ	CAP03.06210	AE	GC/MS	[EPA TO-13A]	Benzo(k)fluoranthene
Certified	Yes	NJ	CAP03.06230	AE	GC/MS	[EPA TO-13A]	Benzo(ghi)perylene
Certified	Yes	NJ	CAP03.06240	AE	GC/MS	[EPA TO-13A]	Chrysene
Certified	Yes	NJ	CAP03.06260	AE	GC/MS	[EPA TO-13A]	Dibenzo(a,h)anthracene
Certified	Yes	NJ	CAP03.06270	AE	GC/MS	[EPA TO-13A]	Fluoranthene
Certified	Yes	NJ	CAP03.06280	AE	GC/MS	[EPA TO-13A]	Fluorene
Certified	Yes	NJ	CAP03.06290	AE	GC/MS	[EPA TO-13A]	Indeno(1,2,3-cd)pyrene
Certified	Yes	NJ	CAP03.06300	AE	GC/MS	[EPA TO-13A]	Naphthalene
Certified	Yes	NJ	CAP03.06320	AE	GC/MS	[EPA TO-13A]	Phenanthrene
Certified	Yes	NJ	CAP03.06330	AE	GC/MS	[EPA TO-13A]	Pyrene
Certified	Yes	NJ	CAP03.06430	AE	GC/MS, CANISTERS	[EPA TO-14A]	Benzene
Certified	Yes	NJ	CAP03.06450	AE	GC/MS, CANISTERS	[EPA TO-14A]	Bromomethane
Certified	Yes	NJ	CAP03.06460	AE	GC/MS, CANISTERS	[EPA TO-14A]	Carbon tetrachloride
Certified	Yes	NJ	CAP03.06470	AE	GC/MS, CANISTERS	[EPA TO-14A]	Chlorobenzene
Certified	Yes	NJ	CAP03.06480	AE	GC/MS, CANISTERS	[EPA TO-14A]	Chloroethane
Certified	Yes	NJ	CAP03.06490	AE	GC/MS, CANISTERS	[EPA TO-14A]	Chloroform
Certified	Yes	NJ	CAP03.06500	AE	GC/MS, CANISTERS	[EPA TO-14A]	Chloromethane
Certified	Yes	NJ	CAP03.06510	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	CAP03.06520	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorobenzene (1,2-)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: CAP03 -- Atmospheric Organic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CAP03.06530	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	CAP03.06540	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	CAP03.06550	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorodifluoromethane
Certified	Yes	NJ	CAP03.06560	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloroethane (1,1-)
Certified	Yes	NJ	CAP03.06570	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloroethane (1,2-)
Certified	Yes	NJ	CAP03.06580	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloroethene (1,1-)
Certified	Yes	NJ	CAP03.06590	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	CAP03.06610	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloropropane (1,2-)
Certified	Yes	NJ	CAP03.06620	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	CAP03.06630	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	CAP03.06640	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorotetrafluoroethane (1,2-)
Certified	Yes	NJ	CAP03.06650	AE	GC/MS, CANISTERS	[EPA TO-14A]	Ethylbenzene
Certified	Yes	NJ	CAP03.06660	AE	GC/MS, CANISTERS	[EPA TO-14A]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	CAP03.06670	AE	GC/MS, CANISTERS	[EPA TO-14A]	Styrene
Certified	Yes	NJ	CAP03.06680	AE	GC/MS, CANISTERS	[EPA TO-14A]	Tetrachloroethane (1,1,2,2-)
Certified	Yes	NJ	CAP03.06690	AE	GC/MS, CANISTERS	[EPA TO-14A]	Tetrachloroethene
Certified	Yes	NJ	CAP03.06700	AE	GC/MS, CANISTERS	[EPA TO-14A]	Toluene
Certified	Yes	NJ	CAP03.06710	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	CAP03.06720	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	CAP03.06730	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trichloroethene
Certified	Yes	NJ	CAP03.06740	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trichlorofluoromethane
Certified	Yes	NJ	CAP03.06760	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trimethylbenzene (1,2,4-)
Certified	Yes	NJ	CAP03.06770	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trimethylbenzene (1,3,5-)
Certified	Yes	NJ	CAP03.06780	AE	GC/MS, CANISTERS	[EPA TO-14A]	Vinyl chloride
Certified	Yes	NJ	CAP03.06790	AE	GC/MS, CANISTERS	[EPA TO-14A]	Xylene (o-)
Certified	Yes	NJ	CAP03.06800	AE	GC/MS, CANISTERS	[EPA TO-14A]	Xylene (m-)
Certified	Yes	NJ	CAP03.06810	AE	GC/MS, CANISTERS	[EPA TO-14A]	Xylene (p-)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SDW02 -- Inorganic Parameters Including Na + Ca

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW02.04000	DW	Ion Chromatography	[EPA 300.0]	Nitrate
Certified	Yes	NJ	SDW02.09000	DW	Spectrophotometric	[SM 4500-NO2 B]	Nitrite
Certified	Yes	NJ	SDW02.14000	DW	Ion Chromatography	[EPA 300.0]	Fluoride
Certified	Yes	NJ	SDW02.15200	DW	Spectrophotometric, Distill, Semi Automated	[EPA 335.4]	Cyanide
Applied	No	NJ	SDW02.19000	DW	Ion Chromatography	[EPA 300.0]	Sulfate
Certified	Yes	NJ	SDW02.20000	DW	ICP	[EPA 200.7]	Sodium
Certified	Yes	NJ	SDW02.27000	DW	ICP	[EPA 200.7]	Calcium
Applied	No	NJ	SDW02.27300	DW	Hardness By Calculation	[EPA 200.7]	Total hardness
Certified	Yes	NJ	SDW02.31000	DW	Ion Chromatography	[EPA 300.0]	Chloride
Certified	Yes	NJ	SDW02.31120	DW	Ion Chromatography	[EPA 314.0]	Perchlorate
Applied	No	NJ	SDW02.31125	DW	LC MS/MS	[EPA 331.0]	Perchlorate
Certified	No	NJ	SDW02.36400	DW	ICP	[EPA 200.7]	Silica

Category: SDW03 -- Analyze-Immediately Inorganic Parameter

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW03.08000	DW	Electrometric	[EPA 150.1]	pH

Category: SDW04 -- Inorganic Parameters, Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW04.03000	DW	ICP	[EPA 200.7]	Aluminum
Certified	Yes	NJ	SDW04.07000	DW	ICP/MS	[EPA 200.8]	Antimony
Certified	Yes	NJ	SDW04.12000	DW	ICP/MS	[EPA 200.8]	Arsenic
Certified	Yes	NJ	SDW04.16000	DW	ICP	[EPA 200.7]	Barium
Certified	Yes	NJ	SDW04.20000	DW	ICP	[EPA 200.7]	Beryllium
Certified	Yes	NJ	SDW04.24000	DW	ICP	[EPA 200.7]	Cadmium
Certified	Yes	NJ	SDW04.28000	DW	ICP	[EPA 200.7]	Chromium
Certified	Yes	NJ	SDW04.33000	DW	ICP	[EPA 200.7]	Copper

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ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
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Category: SDW04 – Inorganic Parameters, Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW04.37000	DW	ICP	[EPA 200.7]	Iron
Certified	Yes	NJ	SDW04.40000	DW	ICP/MS	[EPA 200.8]	Lead
Certified	Yes	NJ	SDW04.44000	DW	ICP	[EPA 200.7]	Manganese
Certified	Yes	NJ	SDW04.46000	DW	Manual Cold Vapor	[EPA 245.1]	Mercury
Certified	Yes	NJ	SDW04.52000	DW	ICP	[EPA 200.7]	Nickel
Certified	Yes	NJ	SDW04.57000	DW	ICP/MS	[EPA 200.8]	Selenium
Certified	Yes	NJ	SDW04.62000	DW	ICP	[EPA 200.7]	Silver
Certified	Yes	NJ	SDW04.65000	DW	ICP/MS	[EPA 200.8]	Thallium
Certified	Yes	NJ	SDW04.67000	DW	ICP	[EPA 200.7]	Zinc

Category: SDW05 – Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW05.12010	DW	Solvent Extract, GC	[EPA 504.1]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	SDW05.12020	DW	Solvent Extract, GC	[EPA 504.1]	Dibromo-3-chloropropane (1,2-)
Certified	Yes	NJ	SDW05.12030	DW	Solvent Extract, GC	[EPA 504.1]	Trichloropropane (1,2,3-)

Category: SDW06 – Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW06.01010	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromoform
Certified	Yes	NJ	SDW06.01020	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chloroform
Certified	Yes	NJ	SDW06.01030	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dibromochloromethane
Certified	Yes	NJ	SDW06.01040	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromodichloromethane
Certified	Yes	NJ	SDW06.02010	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Benzene
Certified	Yes	NJ	SDW06.02020	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Carbon tetrachloride
Certified	Yes	NJ	SDW06.02030	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chlorobenzene
Certified	Yes	NJ	SDW06.02040	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichlorobenzene (1,2-)
Certified	Yes	NJ	SDW06.02050	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichlorobenzene (1,3-)

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Category: SDW06 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW06.02060	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	SDW06.02070	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethane (1,1-)
Certified	Yes	NJ	SDW06.02080	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethane (1,2-)
Certified	Yes	NJ	SDW06.02090	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	SDW06.02100	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethene (trans-1,2-)
Certified	Yes	NJ	SDW06.02110	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methylene chloride (Dichloromethane)
Certified	Yes	NJ	SDW06.02120	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropane (1,2-)
Certified	Yes	NJ	SDW06.02130	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Ethylbenzene
Certified	Yes	NJ	SDW06.02140	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methyl tert-butyl ether
Certified	Yes	NJ	SDW06.02150	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Naphthalene
Certified	Yes	NJ	SDW06.02160	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Styrene
Certified	Yes	NJ	SDW06.02170	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tetrachloroethane (1,1,2,2-)
Certified	Yes	NJ	SDW06.02180	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tetrachloroethene
Certified	Yes	NJ	SDW06.02190	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	SDW06.02200	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichloroethene
Certified	Yes	NJ	SDW06.02210	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Toluene
Certified	Yes	NJ	SDW06.02220	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	SDW06.02230	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethene (1,1-)
Certified	Yes	NJ	SDW06.02240	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichloroethane (1,1,2-)
Certified	Yes	NJ	SDW06.02250	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Vinyl chloride
Certified	Yes	NJ	SDW06.02260	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Xylenes (total)
Certified	Yes	NJ	SDW06.03010	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromobenzene
Certified	Yes	NJ	SDW06.03020	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromochloromethane
Certified	Yes	NJ	SDW06.03030	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromomethane
Certified	Yes	NJ	SDW06.03040	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Butyl benzene (n-)
Certified	Yes	NJ	SDW06.03050	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Sec-butylbenzene
Certified	Yes	NJ	SDW06.03060	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tert-butylbenzene
Certified	Yes	NJ	SDW06.03070	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chloroethane
Certified	Yes	NJ	SDW06.03080	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chloromethane
Certified	Yes	NJ	SDW06.03090	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chlorotoluene (2-)
Certified	Yes	NJ	SDW06.03100	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chlorotoluene (4-)
Certified	Yes	NJ	SDW06.03110	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dibromo-3-chloropropane (1,2-)

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New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SDW06 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW06.03120	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	SDW06.03130	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dibromomethane
Certified	Yes	NJ	SDW06.03140	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichlorodifluoromethane
Certified	Yes	NJ	SDW06.03150	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropane (1,3-)
Certified	Yes	NJ	SDW06.03160	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropane (2,2-)
Certified	Yes	NJ	SDW06.03170	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropene (1,1-)
Certified	Yes	NJ	SDW06.03180	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	SDW06.03190	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	SDW06.03200	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	SDW06.03210	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Isopropylbenzene
Certified	Yes	NJ	SDW06.03220	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Isopropyltoluene (4-)
Certified	Yes	NJ	SDW06.03230	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Propylbenzene (n-)
Certified	Yes	NJ	SDW06.03240	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tetrachloroethane (1,1,1,2-)
Certified	Yes	NJ	SDW06.03250	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichlorobenzene (1,2,3-)
Certified	Yes	NJ	SDW06.03251	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichlorobenzene (1,3,5-)
Certified	Yes	NJ	SDW06.03260	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichlorofluoromethane
Certified	Yes	NJ	SDW06.03270	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichloropropane (1,2,3-)
Certified	Yes	NJ	SDW06.03280	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trimethylbenzene (1,2,4-)
Certified	Yes	NJ	SDW06.03300	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trimethylbenzene (1,3,5-)
Certified	Yes	NJ	SDW06.03310	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Nitrobenzene
Certified	Yes	NJ	SDW06.03410	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Acetone
Certified	Yes	NJ	SDW06.03420	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Acrylonitrile
Certified	Yes	NJ	SDW06.03430	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Allyl chloride
Certified	Yes	NJ	SDW06.03440	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Butanone (2-)
Certified	Yes	NJ	SDW06.03450	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Carbon disulfide
Certified	Yes	NJ	SDW06.03460	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chloroacetonitrile
Certified	Yes	NJ	SDW06.03470	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chlorobutane (1-)
Certified	Yes	NJ	SDW06.03480	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloro-2-butene (trans-1,4-)
Certified	Yes	NJ	SDW06.03490	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropanone (1,1-)
Certified	Yes	NJ	SDW06.03500	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Diethyl ether (Ethyl ether)
Certified	Yes	NJ	SDW06.03510	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Ethyl methacrylate
Certified	Yes	NJ	SDW06.03520	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Hexachloroethane

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Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SDW06 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW06.03530	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Hexanone (2-)
Certified	Yes	NJ	SDW06.03540	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methacrylonitrile
Certified	Yes	NJ	SDW06.03550	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methyl acrylate
Certified	Yes	NJ	SDW06.03560	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methyl iodide
Certified	Yes	NJ	SDW06.03570	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methyl methacrylate
Certified	Yes	NJ	SDW06.03580	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Pentanone (4-methyl-2-)
Certified	Yes	NJ	SDW06.03590	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Nitropropane (2-)
Certified	Yes	NJ	SDW06.03600	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Pentachloroethane
Certified	Yes	NJ	SDW06.03610	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Propionitrile
Certified	Yes	NJ	SDW06.03615	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tert-butyl alcohol
Certified	Yes	NJ	SDW06.03620	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tetrahydrofuran

Category: SHW04 -- Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW04.01000	NPW	Acid Digestion/Surface and Groundwater, ICP, FLAA	[SW-846 3005A, Rev. 1, 7/92]	Metals, Total Rec and Dissolved
Certified	Yes	NJ	SHW04.01500	NPW	Acid Digestion/Aqueous Samples, ICP, FLAA	[SW-846 3010A, Rev. 1, 7/92]	Metals, Total
Applied	No	NJ	SHW04.21000	NPW	Colorimetric	[SW-846 7196A, Rev. 1, 7/92]	Chromium (VI)

Category: SHW05 -- Organic Parameters, Prep. / Screening

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW05.01000	NPW	Separatory Funnel Extraction	[SW-846 3510C, Rev. 3, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.02000	NPW	Continuous Liquid-Liquid Extraction	[SW-846 3520C, Rev. 3, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.07000	NPW	Purge & Trap Aqueous	[SW-846 5030B, Rev. 2, 12/96]	Volatile organics

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Category: SHW09 – Miscellaneous Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW09.17000	NPW	Wheatstone Bridge	[SW-846 9050A, Rev. 1, 12/96]	Specific conductance
Certified	Yes	NJ	SHW09.19000	NPW	Infrared Spectrometry or FID	[SW-846 9060, Rev. 0, 9/86]	Total organic carbon (TOC)

Category: SHW10 – Facility-Specific Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW10.20000	NPW	Facility-Specific	[USER DEFINED RSK-175]	Organics

Category: WPP02 – Inorganic Parameters, Nutrients and Dema

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP02.01500	NPW	Electrometric or Color Titration	[EPA 310.1]	Alkalinity as CaCO3
Certified	Yes	NJ	WPP02.02500	NPW	Distillation, Nesslerization	[EPA 350.2]	Ammonia
Certified	Yes	NJ	WPP02.05000	NPW	Dissolved Oxygen Depletion	[EPA 405.1]	Biochemical oxygen demand
Certified	Yes	NJ	WPP02.06000	NPW	ICP	[EPA 200.7]	Boron
Certified	Yes	NJ	WPP02.08000	NPW	Digestion, ICP	[EPA 200.7]	Calcium
Certified	Yes	NJ	WPP02.09500	NPW	Dissolved Oxygen Depletion, Nitrification Inhibition	[SM 5210 B]	Carbonaceous BOD (CBOD)
Certified	Yes	NJ	WPP02.10500	NPW	Spectrophotometric Manual/Auto	[EPA 410.4]	Chemical oxygen demand
Certified	Yes	NJ	WPP02.12500	NPW	Colorimetric, Automated (Ferricyanide)	[EPA 325.1 OR .2]	Chloride
Certified	Yes	NJ	WPP02.12600	NPW	Ion Chromatography	[EPA 300.0]	Chloride
Certified	Yes	NJ	WPP02.15500	NPW	Distillation, Spectrophotometric (Auto)	[EPA 335.3] [EPA 335.4]	Cyanide
Certified	Yes	NJ	WPP02.18100	NPW	Ion Chromatography	[EPA 300.0]	Fluoride
Certified	Yes	NJ	WPP02.19000	NPW	Titrimetric, EDTA	[EPA 130.2] [SM 2340 B or C]	Hardness - total as CaCO3
Certified	Yes	NJ	WPP02.20100	NPW	Ca + Mg Carbonates, ICP	[EPA 200.7]	Hardness - total as CaCO3
Certified	Yes	NJ	WPP02.21000	NPW	Digestion, Distillation, Nesslerization	[EPA 351.3]	Kjeldahl nitrogen - total
Certified	Yes	NJ	WPP02.24000	NPW	Digestion, ICP	[EPA 200.7]	Magnesium
Certified	Yes	NJ	WPP02.26100	NPW	Ion Chromatography	[EPA 300.0]	Nitrate
Certified	Yes	NJ	WPP02.27000	NPW	Cadmium Reduction, Automated	[EPA 353.2]	Nitrate - nitrite
Certified	Yes	NJ	WPP02.28000	NPW	Spectrophotometric, Manual	[EPA 354.1]	Nitrite

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208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: WPP02 -- Inorganic Parameters, Nutrients and Dema

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP02.30000	NPW	Combustion or Oxidation	[EPA 415.1]	Total organic carbon (TOC)
Certified	Yes	NJ	WPP02.31500	NPW	Ascorbic Acid, Manual Single Reagent	[EPA 365.2]	Orthophosphate
Certified	Yes	NJ	WPP02.35000	NPW	Auto Ascorbic Acid Reduction	[EPA 365.2 + .1]	Phosphorus (total)
Certified	Yes	NJ	WPP02.36500	NPW	Digestion, ICP	[EPA 200.7]	Potassium
Certified	Yes	NJ	WPP02.38000	NPW	Gravimetric, 103-105 Degrees C	[EPA 160.3]	Residue - total
Certified	Yes	NJ	WPP02.38500	NPW	Gravimetric, 180 Degrees C	[EPA 160.1]	Residue - filterable (TDS)
Certified	Yes	NJ	WPP02.39000	NPW	Gravimetric, 103-105 Degrees C, Post Washing	[EPA 160.2]	Residue - nonfilterable (TSS)
Certified	Yes	NJ	WPP02.42500	NPW	0.45u Filtration + ICP	[EPA 200.7]	Silica - dissolved
Certified	Yes	NJ	WPP02.44000	NPW	Digestion, ICP	[EPA 200.7]	Sodium
Certified	Yes	NJ	WPP02.45500	NPW	Wheatstone Bridge	[EPA 120.1]	Specific conductance
Certified	Yes	NJ	WPP02.46500	NPW	Turbidimetric	[EPA 375.4]	Sulfate
Certified	Yes	NJ	WPP02.47100	NPW	Ion Chromatography	[EPA 300.0]	Sulfate
Certified	Yes	NJ	WPP02.48000	NPW	Colorimetric (Methylene Blue)	[EPA 376.2]	Sulfides
Certified	Yes	NJ	WPP02.48500	NPW	Colorimetric (Methylene Blue)	[EPA 425.1]	Surfactants
Certified	Yes	NJ	WPP02.50000	NPW	Nephelometric	[EPA 180.1]	Turbidity

Category: WPP03 -- Analyze-Immediately Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP03.09000	NPW	Electrometric	[EPA 150.1]	pH

Category: WPP04 -- Inorganic Parameters, Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP04.02000	NPW	Digestion, ICP	[EPA 200.7]	Aluminum
Certified	Yes	NJ	WPP04.04500	NPW	Digestion, ICP	[EPA 200.7]	Antimony
Certified	Yes	NJ	WPP04.04600	NPW	ICP/MS	[EPA 200.8]	Antimony
Certified	Yes	NJ	WPP04.05600	NPW	Digestion, ICP	[EPA 200.7]	Arsenic
Certified	Yes	NJ	WPP04.05700	NPW	ICP/MS	[EPA 200.8]	Arsenic

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection
National Environmental Laboratory Accreditation Program
ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS
Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: WPP04 -- Inorganic Parameters, Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP04.08000	NPW	Digestion, ICP	[EPA 200.7]	Barium
Certified	Yes	NJ	WPP04.08200	NPW	ICP/MS	[EPA 200.8]	Barium
Certified	Yes	NJ	WPP04.11000	NPW	Digestion, ICP	[EPA 200.7]	Beryllium
Certified	Yes	NJ	WPP04.11100	NPW	ICP/MS	[EPA 200.8]	Beryllium
Certified	Yes	NJ	WPP04.13500	NPW	Digestion, ICP	[EPA 200.7]	Cadmium
Certified	Yes	NJ	WPP04.13600	NPW	ICP/MS	[EPA 200.8]	Cadmium
Certified	Yes	NJ	WPP04.18000	NPW	Digestion, ICP	[EPA 200.7]	Chromium
Certified	Yes	NJ	WPP04.19500	NPW	Digestion, ICP	[EPA 200.7]	Cobalt
Certified	Yes	NJ	WPP04.19600	NPW	ICP/MS	[EPA 200.8]	Cobalt
Certified	Yes	NJ	WPP04.21500	NPW	Digestion, ICP	[EPA 200.7]	Copper
Certified	Yes	NJ	WPP04.21600	NPW	ICP/MS	[EPA 200.8]	Copper
Certified	Yes	NJ	WPP04.26500	NPW	Digestion, ICP	[EPA 200.7]	Iron
Certified	Yes	NJ	WPP04.28000	NPW	Digestion, ICP	[EPA 200.7]	Lead
Certified	Yes	NJ	WPP04.28100	NPW	ICP/MS	[EPA 200.8]	Lead
Certified	Yes	NJ	WPP04.31000	NPW	Digestion, ICP	[EPA 200.7]	Manganese
Certified	Yes	NJ	WPP04.31100	NPW	ICP/MS	[EPA 200.8]	Manganese
Certified	Yes	NJ	WPP04.33000	NPW	Manual Cold Vapor	[EPA 245.1]	Mercury
Certified	Yes	NJ	WPP04.35000	NPW	Digestion, ICP	[EPA 200.7]	Molybdenum
Certified	Yes	NJ	WPP04.37500	NPW	Digestion, ICP	[EPA 200.7]	Nickel
Certified	Yes	NJ	WPP04.37600	NPW	ICP/MS	[EPA 200.8]	Nickel
Certified	Yes	NJ	WPP04.45500	NPW	Digestion, ICP	[EPA 200.7]	Selenium
Certified	Yes	NJ	WPP04.45600	NPW	ICP/MS	[EPA 200.8]	Selenium
Certified	Yes	NJ	WPP04.48000	NPW	Digestion, ICP	[EPA 200.7]	Silver
Certified	Yes	NJ	WPP04.48200	NPW	ICP/MS	[EPA 200.8]	Silver
Certified	Yes	NJ	WPP04.50000	NPW	Digestion, ICP	[EPA 200.7]	Thallium
Certified	Yes	NJ	WPP04.50100	NPW	ICP/MS	[EPA 200.8]	Thallium
Certified	Yes	NJ	WPP04.51100	NPW	Digestion, ICP	[EPA 200.7]	Tin
Certified	Yes	NJ	WPP04.52050	NPW	Digestion, ICP	[EPA 200.7]	Titanium
Certified	Yes	NJ	WPP04.54000	NPW	Digestion, ICP	[EPA 200.7]	Vanadium
Certified	Yes	NJ	WPP04.54100	NPW	ICP/MS	[EPA 200.8]	Vanadium
Certified	Yes	NJ	WPP04.56500	NPW	Digestion, ICP	[EPA 200.7]	Zinc

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208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: WPP04 -- Inorganic Parameters, Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP04.56600	NPW	ICP/MS	[EPA 200.8]	Zinc

Category: CLP01 -- Multi-Media, Multi-Conc. Inorganics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP01.03002	NPW, SCM	ICP	[EPA ILM05.3]	Aluminum
Certified	Yes	NJ	CLP01.03101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Aluminum
Certified	Yes	NJ	CLP01.06002	NPW, SCM	ICP	[EPA ILM05.3]	Antimony
Certified	Yes	NJ	CLP01.06101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Antimony
Certified	Yes	NJ	CLP01.08002	NPW, SCM	ICP	[EPA ILM05.3]	Arsenic
Certified	Yes	NJ	CLP01.08101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Arsenic
Certified	Yes	NJ	CLP01.11002	NPW, SCM	ICP	[EPA ILM05.3]	Barium
Certified	Yes	NJ	CLP01.11101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Barium
Certified	Yes	NJ	CLP01.14002	NPW, SCM	ICP	[EPA ILM05.3]	Beryllium
Certified	Yes	NJ	CLP01.14101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Beryllium
Certified	Yes	NJ	CLP01.19002	NPW, SCM	ICP	[EPA ILM05.3]	Cadmium
Certified	Yes	NJ	CLP01.19101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Cadmium
Certified	Yes	NJ	CLP01.21002	NPW, SCM	ICP	[EPA ILM05.3]	Calcium
Certified	Yes	NJ	CLP01.24002	NPW, SCM	ICP	[EPA ILM05.3]	Chromium
Certified	Yes	NJ	CLP01.24101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Chromium
Certified	Yes	NJ	CLP01.27002	NPW, SCM	ICP	[EPA ILM05.3]	Cobalt
Certified	Yes	NJ	CLP01.27101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Cobalt
Certified	Yes	NJ	CLP01.30002	NPW, SCM	ICP	[EPA ILM05.3]	Copper
Certified	Yes	NJ	CLP01.30101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Copper
Certified	Yes	NJ	CLP01.33002	NPW, SCM	ICP	[EPA ILM05.3]	Iron
Certified	Yes	NJ	CLP01.36002	NPW, SCM	ICP	[EPA ILM05.3]	Lead
Certified	Yes	NJ	CLP01.36101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Lead
Certified	Yes	NJ	CLP01.38002	NPW, SCM	ICP	[EPA ILM05.3]	Magnesium
Certified	Yes	NJ	CLP01.41002	NPW, SCM	ICP	[EPA ILM05.3]	Manganese
Certified	Yes	NJ	CLP01.41101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Manganese

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208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: CLP01 – Multi-Media, Multi-Conc. Inorganics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP01.42101	NPW, SCM	CVAA, Manual	[EPA ILM05.3]	Mercury
Certified	Yes	NJ	CLP01.47002	NPW, SCM	ICP	[EPA ILM05.3]	Nickel
Certified	Yes	NJ	CLP01.47101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Nickel
Certified	Yes	NJ	CLP01.49002	NPW, SCM	ICP	[EPA ILM05.3]	Potassium
Certified	Yes	NJ	CLP01.51002	NPW, SCM	ICP	[EPA ILM05.3]	Selenium
Certified	Yes	NJ	CLP01.51101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Selenium
Certified	Yes	NJ	CLP01.54002	NPW, SCM	ICP	[EPA ILM05.3]	Silver
Certified	Yes	NJ	CLP01.54101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Silver
Certified	Yes	NJ	CLP01.56002	NPW, SCM	ICP	[EPA ILM05.3]	Sodium
Certified	Yes	NJ	CLP01.59002	NPW, SCM	ICP	[EPA ILM05.3]	Thallium
Certified	Yes	NJ	CLP01.59101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Thallium
Certified	Yes	NJ	CLP01.63002	NPW, SCM	ICP	[EPA ILM05.3]	Vanadium
Certified	Yes	NJ	CLP01.63101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Vanadium
Certified	Yes	NJ	CLP01.66002	NPW, SCM	ICP	[EPA ILM05.3]	Zinc
Certified	Yes	NJ	CLP01.66101	NPW, SCM	ICP/MS	[EPA ILM05.3]	Zinc
Certified	Yes	NJ	CLP01.69101	NPW, SCM	Midi Distillation, Spectrophotometric	[EPA ILM05.3]	Cyanide, Total in Water and Soil / Sediments

Category: CLP02 – Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.01012	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Aldrin
Certified	Yes	NJ	CLP02.01022	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Alpha BHC
Certified	Yes	NJ	CLP02.01032	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Beta BHC
Certified	Yes	NJ	CLP02.01042	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Delta BHC
Certified	Yes	NJ	CLP02.01052	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Lindane (gamma BHC)
Certified	Yes	NJ	CLP02.01062	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Chlordane (alpha)
Certified	Yes	NJ	CLP02.01072	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Chlordane (gamma)
Certified	Yes	NJ	CLP02.01082	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	DDD (4,4'-)
Certified	Yes	NJ	CLP02.01092	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	DDE (4,4'-)
Certified	Yes	NJ	CLP02.01102	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	DDT (4,4'-)

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Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: CLP02 -- Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.01112	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Dieldrin
Certified	Yes	NJ	CLP02.01122	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endosulfan I
Certified	Yes	NJ	CLP02.01132	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endosulfan II
Certified	Yes	NJ	CLP02.01142	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endosulfan sulfate
Certified	Yes	NJ	CLP02.01152	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endrin
Certified	Yes	NJ	CLP02.01162	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endrin aldehyde
Certified	Yes	NJ	CLP02.01172	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endrin ketone
Certified	Yes	NJ	CLP02.01182	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Heptachlor
Certified	Yes	NJ	CLP02.01192	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Heptachlor epoxide
Certified	Yes	NJ	CLP02.01202	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Methoxychlor
Certified	Yes	NJ	CLP02.01212	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Toxaphene
Certified	Yes	NJ	CLP02.01232	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1016
Certified	Yes	NJ	CLP02.01242	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1221
Certified	Yes	NJ	CLP02.01252	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1232
Certified	Yes	NJ	CLP02.01262	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1242
Certified	Yes	NJ	CLP02.01272	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1248
Certified	Yes	NJ	CLP02.01282	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1254
Certified	Yes	NJ	CLP02.01292	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1260
Certified	Yes	NJ	CLP02.01302	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB-1262
Certified	Yes	NJ	CLP02.01312	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB-1268
Certified	Yes	NJ	CLP02.03022	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Benzene
Certified	Yes	NJ	CLP02.03026	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Bromochloromethane
Certified	Yes	NJ	CLP02.03032	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Chlorobenzene
Certified	Yes	NJ	CLP02.03042	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorobenzene (1,2-)
Certified	Yes	NJ	CLP02.03052	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	CLP02.03062	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	CLP02.03066	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dioxane (1,4-)
Certified	Yes	NJ	CLP02.03072	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Ethylbenzene
Certified	Yes	NJ	CLP02.03082	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Isopropylbenzene
Certified	Yes	NJ	CLP02.03088	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorobenzene (1,2,3-)
Certified	Yes	NJ	CLP02.03092	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	CLP02.03102	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Styrene

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Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: CLP02 – Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.03112	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Toluene
Certified	Yes	NJ	CLP02.03116	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Xylene (m- + p-)
Certified	Yes	NJ	CLP02.03118	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Xylene (o-)
Certified	Yes	NJ	CLP02.03142	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Bromodichloromethane
Certified	Yes	NJ	CLP02.03152	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Bromoform
Certified	Yes	NJ	CLP02.03162	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Bromomethane
Certified	Yes	NJ	CLP02.03172	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Carbon tetrachloride
Certified	Yes	NJ	CLP02.03182	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Chloroethane
Certified	Yes	NJ	CLP02.03192	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Chloroform
Certified	Yes	NJ	CLP02.03202	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Chloromethane
Certified	Yes	NJ	CLP02.03212	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	CLP02.03222	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	CLP02.03232	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dibromochloromethane
Certified	Yes	NJ	CLP02.03242	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dibromo-3-chloropropane (1,2-)
Certified	Yes	NJ	CLP02.03252	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorodifluoromethane
Certified	Yes	NJ	CLP02.03262	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethane (1,1-)
Certified	Yes	NJ	CLP02.03272	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethane (1,2-)
Certified	Yes	NJ	CLP02.03282	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethene (1,1-)
Certified	Yes	NJ	CLP02.03292	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethene (trans-1,2-)
Certified	Yes	NJ	CLP02.03302	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	CLP02.03312	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloropropane (1,2-)
Certified	Yes	NJ	CLP02.03322	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	CLP02.03332	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Methylene chloride (Dichloromethane)
Certified	Yes	NJ	CLP02.03342	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Tetrachloroethane (1,1,2,2-)
Certified	Yes	NJ	CLP02.03352	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Tetrachloroethene
Certified	Yes	NJ	CLP02.03362	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	CLP02.03372	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichloroethane (1,1,2-)
Certified	Yes	NJ	CLP02.03382	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichloroethene
Certified	Yes	NJ	CLP02.03392	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorofluoromethane
Certified	Yes	NJ	CLP02.03402	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
Certified	Yes	NJ	CLP02.03412	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Vinyl chloride
Certified	Yes	NJ	CLP02.03432	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Acetone

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COLCHESTER, VT 05446

Category: CLP02 -- Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.03442	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Carbon disulfide
Certified	Yes	NJ	CLP02.03452	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Cyclohexane
Certified	Yes	NJ	CLP02.03462	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Butanone (2-)
Certified	Yes	NJ	CLP02.03472	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Hexanone (2-)
Certified	Yes	NJ	CLP02.03482	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Methyl acetate
Certified	Yes	NJ	CLP02.03492	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Methylcyclohexane
Certified	Yes	NJ	CLP02.03502	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Pentanone (4-methyl-2-)
Certified	Yes	NJ	CLP02.03512	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Tert-butyl methyl ether
Certified	Yes	NJ	CLP02.04022	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Atrazine
Certified	Yes	NJ	CLP02.04032	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	N-Nitrosodiphenylamine
Certified	Yes	NJ	CLP02.04042	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	N-Nitroso-di-n-propylamine
Certified	Yes	NJ	CLP02.04052	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Carbazole
Certified	Yes	NJ	CLP02.04062	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorobenzidine (3,3'-)
Certified	Yes	NJ	CLP02.04072	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chloroaniline (4-)
Certified	Yes	NJ	CLP02.04082	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitroaniline (2-)
Certified	Yes	NJ	CLP02.04092	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitroaniline (3-)
Certified	Yes	NJ	CLP02.04102	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitroaniline (4-)
Certified	Yes	NJ	CLP02.04122	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chloronaphthalene (2-)
Certified	Yes	NJ	CLP02.04132	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Hexachlorobenzene
Certified	Yes	NJ	CLP02.04142	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	CLP02.04152	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Hexachlorocyclopentadiene
Certified	Yes	NJ	CLP02.04162	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Hexachloroethane
Certified	Yes	NJ	CLP02.04182	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bis (2-chloroethoxy) methane
Certified	Yes	NJ	CLP02.04192	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bis (2-chloroisopropyl) ether
Certified	Yes	NJ	CLP02.04202	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bis (2-chloroethyl) ether
Certified	Yes	NJ	CLP02.04212	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chlorophenyl-phenyl ether (4-)
Certified	Yes	NJ	CLP02.04222	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bromophenyl-phenyl ether (4-)
Certified	Yes	NJ	CLP02.04232	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitroaromatics and isophorone
Certified	Yes	NJ	CLP02.04242	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dinitrotoluene (2,4-)
Certified	Yes	NJ	CLP02.04252	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dinitrotoluene (2,6-)
Certified	Yes	NJ	CLP02.04262	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Isophorone
Certified	Yes	NJ	CLP02.04272	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitrobenzene

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: CLP02 – Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.04292	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Butyl benzyl phthalate
Certified	Yes	NJ	CLP02.04302	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bis (2-ethylhexyl) phthalate
Certified	Yes	NJ	CLP02.04312	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Diethyl phthalate
Certified	Yes	NJ	CLP02.04322	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dimethyl phthalate
Certified	Yes	NJ	CLP02.04332	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Di-n-butyl phthalate
Certified	Yes	NJ	CLP02.04342	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Di-n-octyl phthalate
Certified	Yes	NJ	CLP02.04362	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Acenaphthene
Certified	Yes	NJ	CLP02.04372	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Anthracene
Certified	Yes	NJ	CLP02.04382	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Acenaphthylene
Certified	Yes	NJ	CLP02.04392	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(a)anthracene
Certified	Yes	NJ	CLP02.04402	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(a)pyrene
Certified	Yes	NJ	CLP02.04412	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(b)fluoranthene
Certified	Yes	NJ	CLP02.04422	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(ghi)perylene
Certified	Yes	NJ	CLP02.04432	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(k)fluoranthene
Certified	Yes	NJ	CLP02.04442	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chrysene
Certified	Yes	NJ	CLP02.04452	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dibenzo(a,h)anthracene
Certified	Yes	NJ	CLP02.04462	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Fluoranthene
Certified	Yes	NJ	CLP02.04472	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Fluorene
Certified	Yes	NJ	CLP02.04482	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Indeno(1,2,3-cd)pyrene
Certified	Yes	NJ	CLP02.04492	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Methylnaphthalene (2-)
Certified	Yes	NJ	CLP02.04502	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Naphthalene
Certified	Yes	NJ	CLP02.04512	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Phenanthrene
Certified	Yes	NJ	CLP02.04522	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Pyrene
Certified	Yes	NJ	CLP02.04542	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Methyl phenol (4-chloro-3-)
Certified	Yes	NJ	CLP02.04552	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chlorophenol (2-)
Certified	Yes	NJ	CLP02.04562	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorophenol (2,4-)
Certified	Yes	NJ	CLP02.04572	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dimethylphenol (2,4-)
Certified	Yes	NJ	CLP02.04582	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dinitrophenol (2,4-)
Certified	Yes	NJ	CLP02.04592	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dinitrophenol (2-methyl-4,6-)
Certified	Yes	NJ	CLP02.04602	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Methylphenol (2-)
Certified	Yes	NJ	CLP02.04612	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Methylphenol (4-)
Certified	Yes	NJ	CLP02.04622	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitrophenol (2-)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: CLP02 -- Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.04632	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitrophenol (4-)
Certified	Yes	NJ	CLP02.04642	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Pentachlorophenol
Certified	Yes	NJ	CLP02.04652	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Phenol
Certified	Yes	NJ	CLP02.04662	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorophenol (2,4,5-)
Certified	Yes	NJ	CLP02.04672	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorophenol (2,4,6-)
Certified	Yes	NJ	CLP02.04692	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Acetophenone
Certified	Yes	NJ	CLP02.04702	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzaldehyde
Certified	Yes	NJ	CLP02.04712	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Biphenyl (1,1'-)
Certified	Yes	NJ	CLP02.04722	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Caprolactam
Certified	Yes	NJ	CLP02.04732	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dibenzofuran
Certified	Yes	NJ	CLP02.04742	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Tetrachlorobenzene (1,2,4,5-)
Certified	Yes	NJ	CLP02.04752	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Tetrachlorophenol (2,3,4,6-)

Category: SHW02 -- Characteristics of Hazardous Waste

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW02.01000	NPW, SCM	Pensky Martens	[SW-846 1010, Rev. 0, 9/86]	Ignitability
Certified	Yes	NJ	SHW02.06900	NPW, SCM	TCLP, Toxicity Procedure, ZHE	[SW-846 1311, Rev. 0, 7/92]	Volatile organics
Certified	Yes	NJ	SHW02.07000	NPW, SCM	TCLP, Toxicity Procedure, Shaker	[SW-846 1311, Rev. 0, 7/92]	Metals - semi volatile organics
Certified	Yes	NJ	SHW02.08000	NPW, SCM	Synthetic PPT Leachate Procedure	[SW-846 1312, Rev. 0, 9/94]	Metals - organics

Category: SHW03 -- Analyze-Immediately Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW03.01000	NPW, SCM	Aqueous, Electrometric	[SW-846 9040B, Rev. 2, 1/95]	pH

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW04 – Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW04.05000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Aluminum
Applied	No	NJ	SHW04.05500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Aluminum
Certified	Yes	NJ	SHW04.06500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Antimony
Certified	Yes	NJ	SHW04.07000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Antimony
Certified	Yes	NJ	SHW04.09000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Arsenic
Certified	Yes	NJ	SHW04.09500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Arsenic
Certified	Yes	NJ	SHW04.11500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Barium
Certified	Yes	NJ	SHW04.12000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Barium
Certified	Yes	NJ	SHW04.13500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Beryllium
Certified	Yes	NJ	SHW04.14000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Beryllium
Certified	Yes	NJ	SHW04.15100	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Boron
Certified	Yes	NJ	SHW04.15500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Cadmium
Certified	Yes	NJ	SHW04.16000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Cadmium
Certified	Yes	NJ	SHW04.17500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Calcium
Certified	Yes	NJ	SHW04.18500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Chromium
Certified	Yes	NJ	SHW04.19000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Chromium
Certified	Yes	NJ	SHW04.22500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Cobalt
Certified	Yes	NJ	SHW04.23000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Cobalt
Certified	Yes	NJ	SHW04.24500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Copper
Certified	Yes	NJ	SHW04.25000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Copper
Certified	Yes	NJ	SHW04.26000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Iron
Certified	Yes	NJ	SHW04.27500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Lead
Certified	Yes	NJ	SHW04.28000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Lead
Certified	Yes	NJ	SHW04.30500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Magnesium
Certified	Yes	NJ	SHW04.31500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Manganese
Certified	Yes	NJ	SHW04.31600	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Manganese
Certified	Yes	NJ	SHW04.33000	NPW, SCM	AA, Manual Cold Vapor	[SW-846 7470A, Rev. 1, 9/94]	Mercury - liquid waste
Certified	Yes	NJ	SHW04.34000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Molybdenum
Certified	Yes	NJ	SHW04.35500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Nickel
Certified	Yes	NJ	SHW04.36000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 7/92]	Nickel
Certified	Yes	NJ	SHW04.38000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Potassium
Certified	Yes	NJ	SHW04.39000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Selenium

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Laboratory Name: STL BURLINGTON **Laboratory Number:** VT972 **Activity ID:** NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW04 – Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW04.40600	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Selenium
Certified	Yes	NJ	SHW04.41000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Silver
Certified	Yes	NJ	SHW04.41500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Silver
Certified	Yes	NJ	SHW04.43000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Sodium
Certified	Yes	NJ	SHW04.44000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Strontium
Certified	Yes	NJ	SHW04.45000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Thallium
Certified	Yes	NJ	SHW04.45500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Thallium
Certified	Yes	NJ	SHW04.47100	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Tin
Certified	Yes	NJ	SHW04.47500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Vanadium
Certified	Yes	NJ	SHW04.47505	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Vanadium
Certified	Yes	NJ	SHW04.49000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Zinc
Certified	Yes	NJ	SHW04.49500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Zinc

Category: SHW06 -- Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW06.04010	NPW, SCM	GC P&T, FID	[SW-846 8015B, Rev. 2, 12/96]	Gasoline range organic
Certified	Yes	NJ	SHW06.04500	NPW, SCM	Extraction, GC, FID	[SW-846 8015B, Rev. 2, 12/96]	Diesel range organic
Certified	Yes	NJ	SHW06.12010	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Aldrin
Certified	Yes	NJ	SHW06.12020	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Alpha BHC
Certified	Yes	NJ	SHW06.12030	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Beta BHC
Certified	Yes	NJ	SHW06.12040	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Delta BHC
Certified	Yes	NJ	SHW06.12050	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Lindane (gamma BHC)
Certified	Yes	NJ	SHW06.12060	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Chlordane (technical)
Certified	Yes	NJ	SHW06.12070	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Chlordane (alpha)
Certified	Yes	NJ	SHW06.12080	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Chlordane (gamma)
Certified	Yes	NJ	SHW06.12090	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	DDD (4,4'-)
Certified	Yes	NJ	SHW06.12100	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	DDE (4,4'-)
Certified	Yes	NJ	SHW06.12110	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	DDT (4,4'-)
Certified	Yes	NJ	SHW06.12120	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Dieldrin

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection
National Environmental Laboratory Accreditation Program
ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS
Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW06 -- Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW06.12130	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endosulfan I
Certified	Yes	NJ	SHW06.12140	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endosulfan II
Certified	Yes	NJ	SHW06.12150	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endosulfan sulfate
Certified	Yes	NJ	SHW06.12160	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endrin
Certified	Yes	NJ	SHW06.12170	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endrin aldehyde
Certified	Yes	NJ	SHW06.12180	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endrin ketone
Certified	Yes	NJ	SHW06.12190	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Heptachlor
Certified	Yes	NJ	SHW06.12200	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Heptachlor epoxide
Certified	Yes	NJ	SHW06.12210	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Methoxychlor
Certified	Yes	NJ	SHW06.12220	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Toxaphene
Certified	Yes	NJ	SHW06.13110	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1016
Certified	Yes	NJ	SHW06.13120	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1221
Certified	Yes	NJ	SHW06.13130	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1232
Certified	Yes	NJ	SHW06.13140	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1242
Certified	Yes	NJ	SHW06.13150	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1248
Certified	Yes	NJ	SHW06.13160	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1254
Certified	Yes	NJ	SHW06.13170	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1260
Certified	Yes	NJ	SHW06.21010	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Azinphos methyl
Certified	Yes	NJ	SHW06.21015	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Chloropyrifos
Certified	Yes	NJ	SHW06.21020	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Demeton (o-)
Certified	Yes	NJ	SHW06.21030	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Demeton (s-)
Certified	Yes	NJ	SHW06.21060	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Malathion
Certified	Yes	NJ	SHW06.21070	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Parathion ethyl
Certified	Yes	NJ	SHW06.21080	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Parathion methyl
Certified	Yes	NJ	SHW06.23001	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Acifluorfen
Certified	Yes	NJ	SHW06.23003	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Bentazon
Certified	Yes	NJ	SHW06.23005	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Chloramben
Certified	Yes	NJ	SHW06.23010	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	Dalapon
Certified	Yes	NJ	SHW06.23011	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	DCPA
Certified	Yes	NJ	SHW06.23020	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	Dicamba
Certified	Yes	NJ	SHW06.23021	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Dichlorprop
Certified	Yes	NJ	SHW06.23030	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	Dinoseb

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW06 -- Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW06.23040	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	D (2,4-)
Certified	Yes	NJ	SHW06.23041	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	DB (2,4-)
Certified	Yes	NJ	SHW06.23050	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	T (2,4,5-)
Certified	Yes	NJ	SHW06.23060	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	TP (2,4,5-) (Silvex)
Certified	Yes	NJ	SHW06.23061	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Dichlorobenzoic acid (3,5-)
Certified	Yes	NJ	SHW06.23063	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	MCPA
Certified	Yes	NJ	SHW06.23064	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	MCPP
Certified	Yes	NJ	SHW06.23065	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Nitrophenol (4-)
Certified	Yes	NJ	SHW06.23066	NPW, SCM	GC, Extract or Direct Inj, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Pentachlorophenol
Certified	Yes	NJ	SHW06.23070	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	Picloram
Certified	Yes	NJ	SHW06.28010	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	HMX
Certified	Yes	NJ	SHW06.28020	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	RDX
Certified	Yes	NJ	SHW06.28030	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Trinitrobenzene (1,3,5-)
Certified	Yes	NJ	SHW06.28040	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrobenzene (1,3-)
Certified	Yes	NJ	SHW06.28050	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Tetryl
Certified	Yes	NJ	SHW06.28060	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Nitrobenzene
Certified	Yes	NJ	SHW06.28070	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Trinitrotoluene (2,4,6-)
Certified	Yes	NJ	SHW06.28080	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrotoluene (4-amino-2,6-)
Certified	Yes	NJ	SHW06.28090	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrotoluene (2-amino-4,6-)
Certified	Yes	NJ	SHW06.28100	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrotoluene (2,4-)
Certified	Yes	NJ	SHW06.28110	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrotoluene (2,6-)
Certified	Yes	NJ	SHW06.28120	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Nitrotoluene (2-)
Certified	Yes	NJ	SHW06.28130	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Nitrotoluene (3-)
Certified	Yes	NJ	SHW06.28140	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Nitrotoluene (4-)

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04010	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Benzene
Certified	Yes	NJ	SHW07.04011	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromobenzene

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04012	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Butyl benzene (n-)
Certified	Yes	NJ	SHW07.04013	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Sec-butylbenzene
Certified	Yes	NJ	SHW07.04014	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tert-butylbenzene
Certified	Yes	NJ	SHW07.04020	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chlorobenzene
Certified	Yes	NJ	SHW07.04022	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chlorotoluene (2-)
Certified	Yes	NJ	SHW07.04023	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chlorotoluene (4-)
Certified	Yes	NJ	SHW07.04030	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichlorobenzene (1,2-)
Certified	Yes	NJ	SHW07.04040	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	SHW07.04050	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	SHW07.04060	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Ethylbenzene
Certified	Yes	NJ	SHW07.04065	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Isopropylbenzene
Certified	Yes	NJ	SHW07.04067	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Propylbenzene (n-)
Certified	Yes	NJ	SHW07.04070	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Toluene
Certified	Yes	NJ	SHW07.04071	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Isopropyltoluene (4-)
Certified	Yes	NJ	SHW07.04072	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichlorobenzene (1,2,3-)
Certified	Yes	NJ	SHW07.04073	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trimethylbenzene (1,2,4-)
Certified	Yes	NJ	SHW07.04074	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trimethylbenzene (1,3,5-)
Certified	Yes	NJ	SHW07.04080	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Xylenes (total)
Certified	Yes	NJ	SHW07.04081	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Xylene (m-)
Certified	Yes	NJ	SHW07.04082	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Xylene (o-)
Certified	Yes	NJ	SHW07.04083	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Xylene (p-)
Certified	Yes	NJ	SHW07.04088	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Allyl chloride
Certified	Yes	NJ	SHW07.04089	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromochloromethane
Certified	Yes	NJ	SHW07.04090	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromodichloromethane
Certified	Yes	NJ	SHW07.04100	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromoform
Certified	Yes	NJ	SHW07.04110	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromomethane
Certified	Yes	NJ	SHW07.04115	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Butadiene (2-chloro-1,3-)
Certified	Yes	NJ	SHW07.04120	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Carbon tetrachloride
Certified	Yes	NJ	SHW07.04130	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chloroethane
Certified	Yes	NJ	SHW07.04140	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chloroethyl vinyl ether (2-)
Certified	Yes	NJ	SHW07.04150	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chloroform
Certified	Yes	NJ	SHW07.04160	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chloromethane

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New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04170	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	SHW07.04180	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dibromochloromethane
Certified	Yes	NJ	SHW07.04185	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	SHW07.04186	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dibromomethane
Certified	Yes	NJ	SHW07.04187	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dibromo-3-chloropropane (1,2-)
Certified	Yes	NJ	SHW07.04190	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichlorodifluoromethane
Certified	Yes	NJ	SHW07.04200	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethane (1,1-)
Certified	Yes	NJ	SHW07.04210	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethane (1,2-)
Certified	Yes	NJ	SHW07.04220	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethene (1,1-)
Certified	Yes	NJ	SHW07.04230	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethene (trans-1,2-)
Certified	Yes	NJ	SHW07.04235	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	SHW07.04240	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropane (1,2-)
Certified	Yes	NJ	SHW07.04241	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropane (1,3-)
Certified	Yes	NJ	SHW07.04242	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropane (2,2-)
Certified	Yes	NJ	SHW07.04249	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropene (1,1-)
Certified	Yes	NJ	SHW07.04250	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	SHW07.04255	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloro-2-butene (trans-1,4-)
Certified	Yes	NJ	SHW07.04260	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methylene chloride (Dichloromethane)
Certified	Yes	NJ	SHW07.04270	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tetrachloroethane (1,1,2,2-)
Certified	Yes	NJ	SHW07.04280	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tetrachloroethene
Certified	Yes	NJ	SHW07.04282	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tetrahydrofuran
Certified	Yes	NJ	SHW07.04290	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	SHW07.04300	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloroethane (1,1,2-)
Certified	Yes	NJ	SHW07.04310	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloroethene
Certified	Yes	NJ	SHW07.04320	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichlorofluoromethane
Certified	Yes	NJ	SHW07.04322	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
Certified	Yes	NJ	SHW07.04325	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloropropane (1,2,3-)
Certified	Yes	NJ	SHW07.04327	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Vinyl acetate
Certified	Yes	NJ	SHW07.04330	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Vinyl chloride
Certified	Yes	NJ	SHW07.04340	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Acetone
Certified	Yes	NJ	SHW07.04350	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Carbon disulfide
Certified	Yes	NJ	SHW07.04360	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Butanone (2-)

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ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS
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Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
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Category: SHW07 – Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04367	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Ethyl methacrylate
Certified	Yes	NJ	SHW07.04370	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Hexanone (2-)
Certified	Yes	NJ	SHW07.04371	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methacrylonitrile
Certified	Yes	NJ	SHW07.04373	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methyl methacrylate
Certified	Yes	NJ	SHW07.04375	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methyl iodide
Certified	Yes	NJ	SHW07.04376	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Iso-butyl alcohol
Applied	No	NJ	SHW07.04379	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Pentachloroethane
Certified	Yes	NJ	SHW07.04380	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Pentanone (4-methyl-2-)
Certified	Yes	NJ	SHW07.04385	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Propionitrile
Certified	Yes	NJ	SHW07.04390	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methyl tert-butyl ether
Certified	Yes	NJ	SHW07.04395	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tert-butyl alcohol
Applied	No	NJ	SHW07.04398	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Acetonitrile
Certified	Yes	NJ	SHW07.04400	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Acrolein
Certified	Yes	NJ	SHW07.04410	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Acrylonitrile
Certified	Yes	NJ	SHW07.04500	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	SHW07.04540	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260C, Rev. 2, 12/96]	Naphthalene
Certified	Yes	NJ	SHW07.04550	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Styrene
Certified	Yes	NJ	SHW07.04560	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tetrachloroethane (1,1,1,2-)
Certified	Yes	NJ	SHW07.04570	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	SHW07.04590	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dioxane (1,4-)
Certified	Yes	NJ	SHW07.04665	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Acetophenone
Certified	Yes	NJ	SHW07.04670	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Acetylaminofluorene (2-)
Certified	Yes	NJ	SHW07.04675	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Aminobiphenyl (4-)
Certified	Yes	NJ	SHW07.04680	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Aramite
Certified	Yes	NJ	SHW07.04705	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chlorobenzilate
Certified	Yes	NJ	SHW07.04715	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diallate (cis)
Certified	Yes	NJ	SHW07.04720	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diallate (trans)
Certified	Yes	NJ	SHW07.04755	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorophenol (2,6-)
Certified	Yes	NJ	SHW07.04760	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethoate
Certified	Yes	NJ	SHW07.04767	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethylaminoazobenzene
Certified	Yes	NJ	SHW07.04770	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethylbenz(a)anthracene (7,12-)
Certified	Yes	NJ	SHW07.04775	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethyl benzidine (3,3-)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04795	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Famphur
Certified	Yes	NJ	SHW07.04805	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachloropropene
Certified	Yes	NJ	SHW07.04810	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Isodrin
Certified	Yes	NJ	SHW07.04815	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Isosafrole (cis-)
Certified	Yes	NJ	SHW07.04820	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Isosafrole (trans-)
Certified	Yes	NJ	SHW07.04830	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methanesulfonate (Ethyl-)
Certified	Yes	NJ	SHW07.04835	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methanesulfonate (Methyl-)
Certified	Yes	NJ	SHW07.04840	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methapyrilene
Certified	Yes	NJ	SHW07.04845	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylcholanthrene (3-)
Certified	Yes	NJ	SHW07.04850	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Napthoquinone (1,4-)
Certified	Yes	NJ	SHW07.04855	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Napththylamine (1-)
Certified	Yes	NJ	SHW07.04860	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Napththylamine (2-)
Certified	Yes	NJ	SHW07.04870	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitroso-di-n-butylamine
Certified	Yes	NJ	SHW07.04875	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosomorpholine
Certified	Yes	NJ	SHW07.04880	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosopiperidine
Certified	Yes	NJ	SHW07.04885	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Parathion
Certified	Yes	NJ	SHW07.04890	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Parathion methyl
Certified	Yes	NJ	SHW07.04895	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pentachlorobenzene
Certified	Yes	NJ	SHW07.04900	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pentachloroethane
Certified	Yes	NJ	SHW07.04905	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pentachloronitrobenzene
Certified	Yes	NJ	SHW07.04910	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phenacetin
Certified	Yes	NJ	SHW07.04920	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phenylethylamine (alpha, alpha-Dimethyl)
Certified	Yes	NJ	SHW07.04925	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phorate
Certified	Yes	NJ	SHW07.04930	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phosphorothioate (O,O,O-Triethyl)
Certified	Yes	NJ	SHW07.04940	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Picoline (2-)
Certified	Yes	NJ	SHW07.04945	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pronamide
Certified	Yes	NJ	SHW07.04950	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Quinoline -1-Oxide (4-Nitro)
Certified	Yes	NJ	SHW07.04955	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Safrole
Certified	Yes	NJ	SHW07.04960	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8270C, Rev. 3, 12/96]	Sulfotepp
Certified	Yes	NJ	SHW07.04975	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Tetrachlorobenzene (1,2,4,5-)
Certified	Yes	NJ	SHW07.04980	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Tetrachlorophenol (2,3,4,6-)
Certified	Yes	NJ	SHW07.04985	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Toluidine (2-) (2-Methylaniline)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW07 – Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04990	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Toluidine (5-Nitro-2-)
Certified	Yes	NJ	SHW07.05004	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosodiethylamine
Certified	Yes	NJ	SHW07.05005	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosodimethylamine
Certified	Yes	NJ	SHW07.05006	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitroso-di-n-propylamine
Certified	Yes	NJ	SHW07.05010	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosodiphenylamine
Certified	Yes	NJ	SHW07.05011	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosomethylethylamine
Certified	Yes	NJ	SHW07.05012	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosopyrrolidine
Certified	Yes	NJ	SHW07.05020	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diphenylamine
Certified	Yes	NJ	SHW07.05030	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Carbazole
Certified	Yes	NJ	SHW07.05038	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzidine
Certified	Yes	NJ	SHW07.05040	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorobenzidine (3,3'-)
Certified	Yes	NJ	SHW07.05045	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diphenylhydrazine (1,2-)
Certified	Yes	NJ	SHW07.05048	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Aniline
Certified	Yes	NJ	SHW07.05050	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chloraniline (4-)
Certified	Yes	NJ	SHW07.05060	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitroaniline (2-)
Certified	Yes	NJ	SHW07.05062	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitroaniline (3-)
Certified	Yes	NJ	SHW07.05063	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitroaniline (4-)
Certified	Yes	NJ	SHW07.05070	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chloronaphthalene (2-)
Certified	Yes	NJ	SHW07.05080	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachlorobenzene
Certified	Yes	NJ	SHW07.05090	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	SHW07.05100	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachlorocyclopentadiene
Certified	Yes	NJ	SHW07.05110	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachloroethane
Certified	Yes	NJ	SHW07.05120	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	SHW07.05130	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bis (2-chloroethoxy) methane
Certified	Yes	NJ	SHW07.05132	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bis (2-chloroethyl) ether
Certified	Yes	NJ	SHW07.05140	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bis (2-chloroisopropyl) ether
Certified	Yes	NJ	SHW07.05150	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chlorophenyl-phenyl ether (4-)
Certified	Yes	NJ	SHW07.05160	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bromophenyl-phenyl ether (4-)
Certified	Yes	NJ	SHW07.05170	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dinitrotoluene (2,4-)
Certified	Yes	NJ	SHW07.05180	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dinitrotoluene (2,6-)
Certified	Yes	NJ	SHW07.05190	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Isophorone
Certified	Yes	NJ	SHW07.05200	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitrobenzene

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.05210	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Butyl benzyl phthalate
Certified	Yes	NJ	SHW07.05220	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bis (2-ethylhexyl) phthalate
Certified	Yes	NJ	SHW07.05230	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diethyl phthalate
Certified	Yes	NJ	SHW07.05240	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethyl phthalate
Certified	Yes	NJ	SHW07.05250	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Di-n-butyl phthalate
Certified	Yes	NJ	SHW07.05260	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Di-n-octyl phthalate
Certified	Yes	NJ	SHW07.05270	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Acenaphthene
Certified	Yes	NJ	SHW07.05280	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Anthracene
Certified	Yes	NJ	SHW07.05290	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Acenaphthylene
Certified	Yes	NJ	SHW07.05300	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(a)anthracene
Certified	Yes	NJ	SHW07.05310	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(a)pyrene
Certified	Yes	NJ	SHW07.05320	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(b)fluoranthene
Certified	Yes	NJ	SHW07.05330	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(ghi)perylene
Certified	Yes	NJ	SHW07.05340	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(k)fluoranthene
Certified	Yes	NJ	SHW07.05350	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chrysene
Certified	Yes	NJ	SHW07.05360	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dibenzo(a,h)anthracene
Certified	Yes	NJ	SHW07.05370	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Fluoranthene
Certified	Yes	NJ	SHW07.05380	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Fluorene
Certified	Yes	NJ	SHW07.05390	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Indeno(1,2,3-cd)pyrene
Certified	Yes	NJ	SHW07.05400	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylnaphthalene (2-)
Certified	Yes	NJ	SHW07.05410	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Naphthalene
Certified	Yes	NJ	SHW07.05420	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phenanthrene
Certified	Yes	NJ	SHW07.05430	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pyrene
Certified	Yes	NJ	SHW07.05440	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methyl phenol (4-chloro-3-)
Certified	Yes	NJ	SHW07.05450	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chlorophenol (2-)
Certified	Yes	NJ	SHW07.05460	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorophenol (2,4-)
Certified	Yes	NJ	SHW07.05470	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethylphenol (2,4-)
Certified	Yes	NJ	SHW07.05480	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dinitrophenol (2,4-)
Certified	Yes	NJ	SHW07.05490	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dinitrophenol (2-methyl-4,6-)
Certified	Yes	NJ	SHW07.05500	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylphenol (2-)
Certified	Yes	NJ	SHW07.05510	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylphenol (4-)
Certified	Yes	NJ	SHW07.05520	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitrophenol (2-)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW07 – Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.05530	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitrophenol (4-)
Certified	Yes	NJ	SHW07.05540	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pentachlorophenol
Certified	Yes	NJ	SHW07.05550	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phenol
Certified	Yes	NJ	SHW07.05560	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Trichlorophenol (2,4,5-)
Certified	Yes	NJ	SHW07.05570	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Trichlorophenol (2,4,6-)
Certified	Yes	NJ	SHW07.05590	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylphenol (3-)
Certified	Yes	NJ	SHW07.05600	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dibenzofuran
Certified	Yes	NJ	SHW07.05691	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorobenzene (1,2-)
Certified	Yes	NJ	SHW07.05692	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	SHW07.05700	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	SHW07.05710	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzoic acid
Certified	Yes	NJ	SHW07.05720	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzyl alcohol
Certified	Yes	NJ	SHW07.05750	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pyridine

Category: SHW09 – Miscellaneous Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW09.05000	NPW, SCM	Colorimetric, Automated	[SW-846 9012A, Rev. 1, 12/96]	Cyanide
Certified	Yes	NJ	SHW09.09000	NPW, SCM	Redox Titration	[SW-846 9030B, Rev. 2, 12/96]	Sulfides, acid sol. & insol.
Certified	Yes	NJ	SHW09.10100	NPW, SCM	Titration	[SW-846 9034, Rev. 0, 12/96]	Sulfides, acid sol. & insol.
Certified	Yes	NJ	SHW09.13000	NPW, SCM	Turbidimetric	[SW-846 9038, Rev. 0, 9/86]	Sulfate
Certified	Yes	NJ	SHW09.13050	NPW, SCM	Ion Chromatography	[SW-846 9056, Rev. 0, 9/94]	Sulfate
Certified	Yes	NJ	SHW09.14000	NPW, SCM	Electrometric	[SW-846 9040B, Rev. 2, 1/95]	pH - waste, >20% water
Applied	No	NJ	SHW09.18010	NPW, SCM	Ion Chromatography, Bomb Combustion, Solids	[SW-846 9056, Rev. 0, 9/94]	Inorganic anions
Certified	Yes	NJ	SHW09.21000	NPW, SCM	Colorimetric, Man, 4AAP Distillation	[SW-846 9065, Rev. 0, 9/86]	Phenols
Certified	Yes	NJ	SHW09.30150	NPW, SCM	Ion Chromatography	[SW-846 9056, Rev. 0, 12/94]	Nitrate
Certified	Yes	NJ	SHW09.31000	NPW, SCM	Colorimetric, Automated (Ferri-CN AAI)	[SW-846 9250, Rev. 0, 9/86]	Chloride
Certified	Yes	NJ	SHW09.33100	NPW, SCM	Ion Chromatography	[SW-846 9056, Rev. 0, 12/96]	Chloride
Certified	Yes	NJ	SHW09.34150	NPW, SCM	Ion Chromatography	[SW-846 9056, Rev. 0, 12/96]	Fluoride

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW04 -- Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW04.03000	SCM	Acid Digestion, Soil Sediment & Sludge	[SW-846 3050B, Rev. 2, 12/96]	Metals
Certified	Yes	NJ	SHW04.33500	SCM	AA, Manual Cold Vapor	[SW-846 7471A, Rev. 1, 9/94]	Mercury - solid waste

Category: SHW05 -- Organic Parameters, Prep. / Screening

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW05.03000	SCM	Soxhlet Extraction	[SW-846 3540C, Rev. 3, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.04000	SCM	Automatic Soxhlet Extraction	[SW-846 3541, Rev. 0, 9/94]	Semivolatile organics
Certified	Yes	NJ	SHW05.05000	SCM	Ultrasonic Extraction	[SW-846 3550B, Rev. 2, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.06000	SCM	Waste Dilution	[SW-846 3580A, Rev. 1, 7/92]	Organics
Certified	Yes	NJ	SHW05.07300	SCM	Closed System Purge & Trap	[SW-846 5035, Rev. 0, 12/96]	Volatile organics - low conc.
Certified	Yes	NJ	SHW05.07310	SCM	Methanol Extract, Closed System P & T	[SW-846 5035, Rev. 0 12/96]	Volatile organics - high conc.
Certified	Yes	NJ	SHW05.12000	SCM	Cleanup-Florisil	[SW-846 3620B, Rev. 2, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.13000	SCM	Cleanup-Silica Gel	[SW-846 3630C, Rev. 3, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.14000	SCM	Cleanup-Gel Permeation	[SW-846 3640A, Rev. 1, 9/94]	Semivolatile organics
Applied	No	NJ	SHW05.15000	SCM	Cleanup-Acid/Base Partition	[SW-846 3650B, Rev. 2, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.16000	SCM	Cleanup-Sulfur Removal	[SW-846 3660B, Rev. 2, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.17000	SCM	Cleanup-Sulfuric Acid/KMnO4	[SW-846 3665A, Rev. 1, 12/96]	Semivolatile organics
Applied	No	NJ	SHW05.18000	SCM	Headspace, GC or GC/MS Screen	[SW-846 3810, Rev. 0, 9/86]	Volatile organics

Category: SHW09 -- Miscellaneous Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW09.16000	SCM	Mix with Water or Calcium Chloride	[SW-846 9045C, Rev. 3, 1/95]	pH - soil and waste
Applied	No	NJ	SHW09.40000	SCM	Soils, Sodium Acetate	[SW-846 9081, Rev. 0, 9/86]	Cation-exchange capacity

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 03/24/2006 until 06/30/2006

Laboratory Name: STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC050009
208 SOUTH PARK DR
STE 1
COLCHESTER, VT 05446

Category: SHW10 -- Facility-Specific Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW10.30025	SCM	Ion Chromatography	[USER DEFINED EPA 314.0, Mod.]	Perchlorate in Soils

Joseph F. Aiello, Chief

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Appendix B

Laboratory Standard Operating Procedures (are included on CD only)

Appendix C

Anticipated Sampling Grids and Associated Calculations

INDEPENDENT VERIFICATION SAMPLING CALCULATIONS – REFUSE AREA AND OXBOW AREA

TIME CRITICAL REMOVAL ACTION WORK PLAN
ALLIED PAPER, INC./PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE
GEORGIA-PACIFIC CORPORATION
KALAMAZOO, MICHIGAN

Introduction

Verification sampling is to be preformed at the Refuse and Oxbow Areas as part of the paper-making residuals (residuals) removal activities. Verification sampling frequency requirements were determined based on the *Sampling Strategies and Statistics Training Materials for Part 201Cleanup Criteria* (MDEQ Guidance Document; MDEQ 2004). Independent sampling frequency and location requirements were developed for both the Refuse and Oxbow Areas as these are regarded as separate "sites" in relation to the MDEQ Guidance Document. A sampling strategy that facilitated the selection of unbiased sampling locations using girding was used, pursuant to the MDEQ Guidance Document.

Size of "Site"

As described in the MDEQ Guidance Document, the verification sampling frequency and locations are based on the planimetric area to be remediated, or, as designated in the MDEQ Guidance Document, the size of the "site". Determination of the "site" size includes calculating the combined area of the excavation sidewalls and base. This calculation, and a discussion of the "site" size based on the MDEQ Guidance Document, is presented below.

Determining the Appropriate Grid Interval

In accordance with MDEQ Guidance Document, the grid interval to be established for verification sample collection is determined based on "site" size (i.e., small, medium or large), and the corresponding total "site" area (sidewall plus base areas). The grid interval for a medium and large-size "site" is calculated using the following equations:

Medium Site $$\frac{\sqrt{A/\pi}}{4} = G.I.$$ Large Site $$\sqrt{\frac{A/\pi}{SF}} = G.I.$$

where:

G.I. = Grid Interval
A = "Site" Area; and
π = Pi (3.14).

Calculation of the grid interval for both the Refuse and Oxbow Areas are presented below.

Refuse Area

The total area of the Refuse Area excavation, including excavation sidewalls and base, is approximately 103,179 square feet (ft^2), thus utilizing the medium site size equation above, the grid interval equals 45 ft. Utilizing a 45-foot grid spacing to establish the verification sample collection locations results in 11 grid stations located within and along the sidewalls of the removal area.

Oxbow Area

The total area of the Oxbow Area excavation, including excavation sidewalls and base, is approximately 173,400 ft^2, thus utilizing the large site size equation above, the grid interval equals 30 ft. Utilizing a 30-foot grid spacing to establish the verification sample collection locations results in 189 grid stations located within and along the sidewalls of the removal area.

Estimating the Number of Samples to be collected on the Established Grid

As recommended in the MDEQ Guidance Document, a minimum of 9 samples or 25 percent of the total number of grid stations, whichever is larger, should be collected and analyzed as part of the verification sampling program. Applying this guidance information to the Refuse and Oxbow removal areas, and assuming grid intervals of 45 feet (ft), and 30 ft, respectively, the appropriate number of verification samples is determined as described below.

The sample collection requirements for the Refuse and Oxbow excavations are calculated based on the respective areas of the sidewall and base, and the sampling frequency criteria presented in the MDEQ Guidance Document. The table below presents calculations for the Refuse and Oxbow excavations. These calculations are based on the excavation areas presented above, under the *size of the "site"* section.

Removal Area	**Grid Station Area** (ft^2)	**Total Sidewall Area** (ft^2)	**Total Base Area** (ft^2)	**Number of Grid Stations**	**25% of Grid Stations**	**Minimum Number of Samples**
Refuse Area	2,025	11,726	91,453	45	11.25	11
Oxbow Area	900	3,272	170,128	189	47.25	47
					Total Samples	**58**

Based on the above calculations, a minimum number of 11, and 47 samples should be taken from each of the Refuse Area and Oxbow Area excavations, respectively, for a total of 58 samples.

CLIENT: Georgia-Pacific Corporation **PROJECT:** Georgia-Pacific Corporation Kalamazoo Mill and Former Hawthorne Mill Properties
TITLE: Sample Calculations **Prepared By:** D.O.K. **Date:** June 2005
SUBJECT: Verification Sampling Calculations – Refuse Area and Oxbow Area **Checked By:** D.J.H **Date:** June 2005

OBJECTIVE:

Determine the frequency of post-excavation verification samples required for the Refuse Area Removal Area and the Oxbow Area Removal Area of the Georgia-Pacific Corporation (Georgia-Pacific) Kalamazoo Mill Property (Kalamazoo Mill Property) and the former Hawthorne Mill Property (Hawthorne Mill Property), respectively.

REFERENCES:

1. MDEQ. 2002. *Sampling Strategies and Statistics Training Materials for Part 201 Cleanup Criteria* (MDEQ. April 14, 2004) Remediation and Redevelopment Division (MDEQ Guidance Document; Lansing, MI: 2002).

ASSUMPTIONS:

1. The removal areas were determined from the approximate Refuse Area Removal Area and Oxbow Area Removal Area limits on Figure 1 of the A*ction Removal Area Work Plan* (Work Plan; BBL, 2005). The Refuse Area Removal Area equaled approximately 2 acres and the Oxbow Area Removal Area equaled approximately 4 acres.

2. The perimeter of the assumed excavation areas was determined from the approximate Refuse Area Removal Area and Oxbow Area Removal Area limits on Figure 1 of the Work Plan. The Refuse Area Removal Area perimeter equaled approximately 1,303 square feet (ft^2) and the Oxbow Area Removal Area perimeter equaled approximately 1,636 ft^2.

3. The Refuse Area and Oxbow Area excavation depths were assumed to be 9 feet (ft) and 2 ft, respectively, in accordance with the Work Plan.

4. The site factor (S.F.) for the Oxbow Area Removal Area was determined from the approximate Oxbow Area Removal Area limits on Figure 1 of the Work Plan.

CALCULATIONS:

Sampling Grid Interval Calculations

Consistent with the MDEQ Guidance Document the Refuse Area Removal Area is characterized as a medium site (i.e., an excavation area between 0.25 and 3.0 acres) and the Oxbow Area Removal Area is characterized as a large site (i.e., an excavation area greater than 3.0 acres), as such, the grid interval shall be calculated using the following equations:

Medium Site $$\frac{\sqrt{A/\pi}}{4} = \text{G.I.}$$

Large Site $$\sqrt{\frac{A * \pi}{SF}} = \text{G.I.}$$

where,

G.I. = Grid interval.
A = Area to be grid (ft^2. The area equals the sum of the excavation base and sidewalls areas).

CLIENT: Georgia-Pacific Corporation PROJECT: Georgia-Pacific Corporation Kalamazoo Mill and Former Hawthorne Mill Properties
TITLE: Sample Calculations Prepared By: D.O.K. Date: June 2005
SUBJECT: Verification Sampling Calculations – Refuse Area and Oxbow Area Checked By: D.J.H Date: June 2005

S.F. = Site factor, length of area to be grid (unit less).

Refuse Area Removal Area

Consistent with the MDEQ Guidance Document, the grid interval for a medium site is given by Equation 1. The excavation base area equaled 91,453 ft^2. The sidewall area was calculated by multiplying the length of the perimeter of the assumed excavation area by the depth of the excavation, and is given by the following:

$$A_{sidewall} = 1{,}303\,\text{ft} * 9\,\text{ft} = 11{,}726\,\text{ft}^2$$

The total area was calculated as the sum of the excavation base and sidewalls, as follows:

$$A_{Total} \cong 103{,}179\,\text{ft}^2 \cong 2.37\,\text{acres}$$

$$\text{G.I.} = \frac{\sqrt{103{,}179\,\text{ft}^2 / \pi}}{4} = 45\,\text{ft}$$

Assume a 45 ft gird interval, as such, the number of nodes was determined by:

$$91{,}453\ ft^2 /(45\ ft * 45\ ft) = 45\, nodes$$

Consistent with the Guidance Document, the minimum number of samples was determined to be the greater of 9 samples or 25% of the number of nodes:

$$45\ \text{nodes} * 0.25 = 11.25$$

A minimum of 11 post-excavation samples will be taken within the Refuse Area.

Oxbow Area Removal Area

Consistent with the MDEQ Guidance Document, the grid interval for a large site is given by Equation 2. The excavation base area equaled 170,128 ft^2. The sidewall area was calculated by multiplying the length of the perimeter of the assumed excavation area by the assumed depth of the excavation, and is given by the following:

$$A_{sidewall} = 1{,}636\,\text{ft} * 2\,\text{ft} = 3{,}272\,\text{ft}^2$$

The total area was calculated as the sum of the excavation base and sidewalls, as follows:

$$A_{Total} \cong 173{,}400\,\text{ft}^2 \cong 3.98\,\text{acres}$$

CLIENT: Georgia-Pacific Corporation **PROJECT:** Georgia-Pacific Corporation Kalamazoo Mill and Former Hawthorne Mill Properties
TITLE: Sample Calculations **Prepared By:** D.O.K. **Date:** June 2005
SUBJECT: Verification Sampling Calculations – Refuse Area and Oxbow Area **Checked By:** D.J.H **Date:** June 2005

$$\text{G.I.} = \sqrt{\frac{173{,}400\,\text{ft} * \pi}{635}} = 29.29\,\text{ft}$$

Assume a 30 ft gird interval, as such, the approximate number of nodes was determined by:

$$170{,}128\,\text{ft}^2 /(30\,\text{ft} * 30\,\text{ft}) = 189\,\text{nodes}$$

Consistent with the Guidance Document, the minimum number of samples was determined to be the greater of 9 samples or 25% of the number of nodes:

$$189\ \text{nodes} * 0.25 = 47.25 = 47$$

A minimum of 47 post-excavation samples will be taken within the Oxbow Area.

SUMMARY:

Based on the above calculations, a minimum number of 11 and 47 samples should have been taken from the Refuse Area and Oxbow Area excavations, respectively, for a total of 58 samples.

Appendix D

Particulate Monitoring Standard Operating Procedures

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Standard Operating Procedure: Particulate Monitoring

I. Scope and Application

The objective of this Standard Operating Procedure (SOP) is to describe the procedures necessary to monitor the active work area for airborne particulate concentrations using a portable particulate monitor (e.g., MIE MiniRAM, MIE PDR1200, or equivalent). This SOP describes equipment, field procedures, materials, and documentation procedures.

This SOP may be varied or changed, as required, depending on site conditions.

II. Personnel Qualifications

Personnel will have current health and safety training, including 40-hour HAZWOPER training, site-specific training, first aid and CPR, and site supervisor training, as needed. In addition, personnel will be versed in the relevant SOPs and possess the required skills and experience necessary to successfully complete the desired field work.

III. Equipment List

The following materials, as required, shall be available when performing dust monitoring:

- Appropriate personal protective equipment (PPE) as specified in the site *Health and Safety Plan* (HASP; BBL, 2006);
- Particulate monitor and operating manual;
- Extra 9 volt batteries;
- Attachable personal-type pump unit – depending on the model used;
- Hand-inflatable "zero air" pouch or zeroing filter cartridge – depending on the model used;
- Field calibration log; and
- Field notebook

IV. Cautions

Take care to not subject the particulate monitoring device to excessive shock, vibration, temperature or humidity. If the unit has been exposed to low temperatures (e.g. in the trunk of a car during winter) for more than a few minutes, care should be taken to allow the instrument to return near room temperature before operating it indoors. This is advisable because water vapor may condense on the interior surfaces of the unit causing temporary malfunction or erroneous readings. Direct access of light to the sensing chamber should also be avoided.

V. Health and Safety Considerations

Health and safety considerations are discussed in the site HASP (BBL, 2006)

VI. Procedure

General steps to operate an MIE PDR1200 particulate monitor are discussed below; however, the owner's manual should be consulted prior to use to ensure that the device is operated correctly, especially in the event that a different but equivalent device is chosen.

- Zero the unit by connecting the filter cartridge to the cyclone inlet and running the pump for at least one minute, until the CALIBRATION: OK message appears on the PDR-1200 display.
- Disconnect the filter cartridge from the cyclone inlet.
- Ensure the 37 millimeter filter holder is installed on the left side of the sensing chamber prior to connecting the pump;
- The unit can now be configured to run.

VII. Waste Management

Not applicable.

VIII. Data Recording and Management

Measurements will be recorded in the field notebook at the time of measurement with notation of date, time, and location. If a data memory is available, readings will be downloaded from the unit upon access to a computer with software to retrieve the data.

IX. Quality Assurance

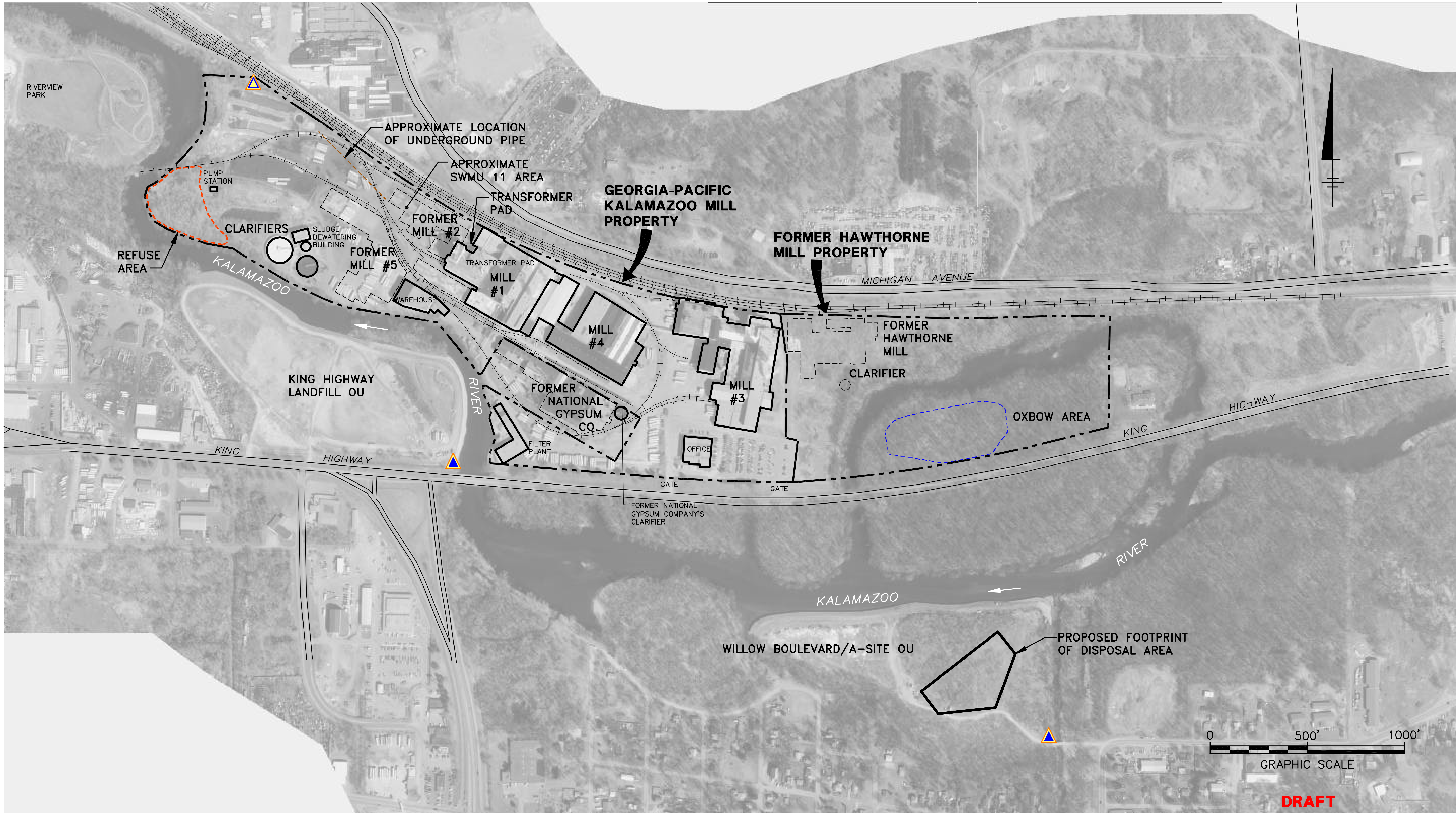
General maintenance procedures associated with the particulate monitor are discussed below and detailed in the manufacturer's instructions; which should be consulted to verify accuracy, since a similar, though functional equivalent, device may be utilized.

Unless a MALFUNCTION message is displayed, or other operational problems occur, the unit should be returned to the factory once every two years for routine check out, test, cleaning and calibration check.

The optical sensing chamber should be cleaned whenever the unit alerts the user with a BACKGROUND HIGH message. The cyclone (if using an MIE PDR1200) should also be cleaned concurrent with the optical sensing chamber.

X. References

Blasland, Bouck & Lee, Inc. 2006. *Time Critical Removal Action for the Refuse Area at the Georgia-Pacific Corporation Kalamazoo Mill Property and the Oxbow Area at the Former Hawthorne Mill Property – Health and Safety Plan – Allied Paper inc./Portage Creek/Kalamazoo River Superfund Site*.



NOTES:

1. PLANIMETRIC MAPPING, INCLUDING PROPERTY BOUNDARIES, IS APPROXIMATE.
2. AERIAL IMAGE DERIVED FROM ORTHOPHOTOGRAPHIC DATA BY AIR LAND SURVEYS, INC., FLOWN 4/24/99.
3. PROPOSED MONITORING LOCATIONS ARE APPROXIMATE, LOCATIONS MAY VARY BASED ON FIELD CONDITIONS ENCOUNTERED DURING THE REMOVAL ACTION.

LEGEND:

- APPROXIMATE REFUSE AREA REMOVAL AREA
- APPROXIMATE BOUNDARY OF KALAMAZOO MILL AND HAWTHORNE MILL PROPERTIES
- APPROXIMATE BOUNDARY OF FORMER MILLS
- APPROXIMATE OXBOW AREA REMOVAL AREA
- PROPOSED AIR MONITORING LOCATION AND PCB ACTION LEVEL OF 0.2 ug/m³
- PROPOSED AIR MONITORING LOCATION AND PCB ACTION LEVEL OF 0.02 ug/m³

GEORGIA-PACIFIC CORPORATION
KALAMAZOO MILL PROPERTY
REMOVAL ACTION WORK PLAN

GEORGIA-PACIFIC KALAMAZOO MILL AND HAWTHORNE MILL SITE PLAN

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FIGURE
1

SYR-85-RLP RCA LJP LAYER: ON=*, OFF=*REF*, [DGTZD-LAGOON, [STREET-NAMES
F:\ACTIVE\-DWG\ACT\64585675\RAWP\64585B02.DWG SAVED:6/30/2006 9:09 AM LAYOUT:Layout1 PAGESETUP:---- PENTABLE:PLTFULL.CTB PRINTED:6/30/2006 9:18 AM BY:LJP
PROJECTNAME:----
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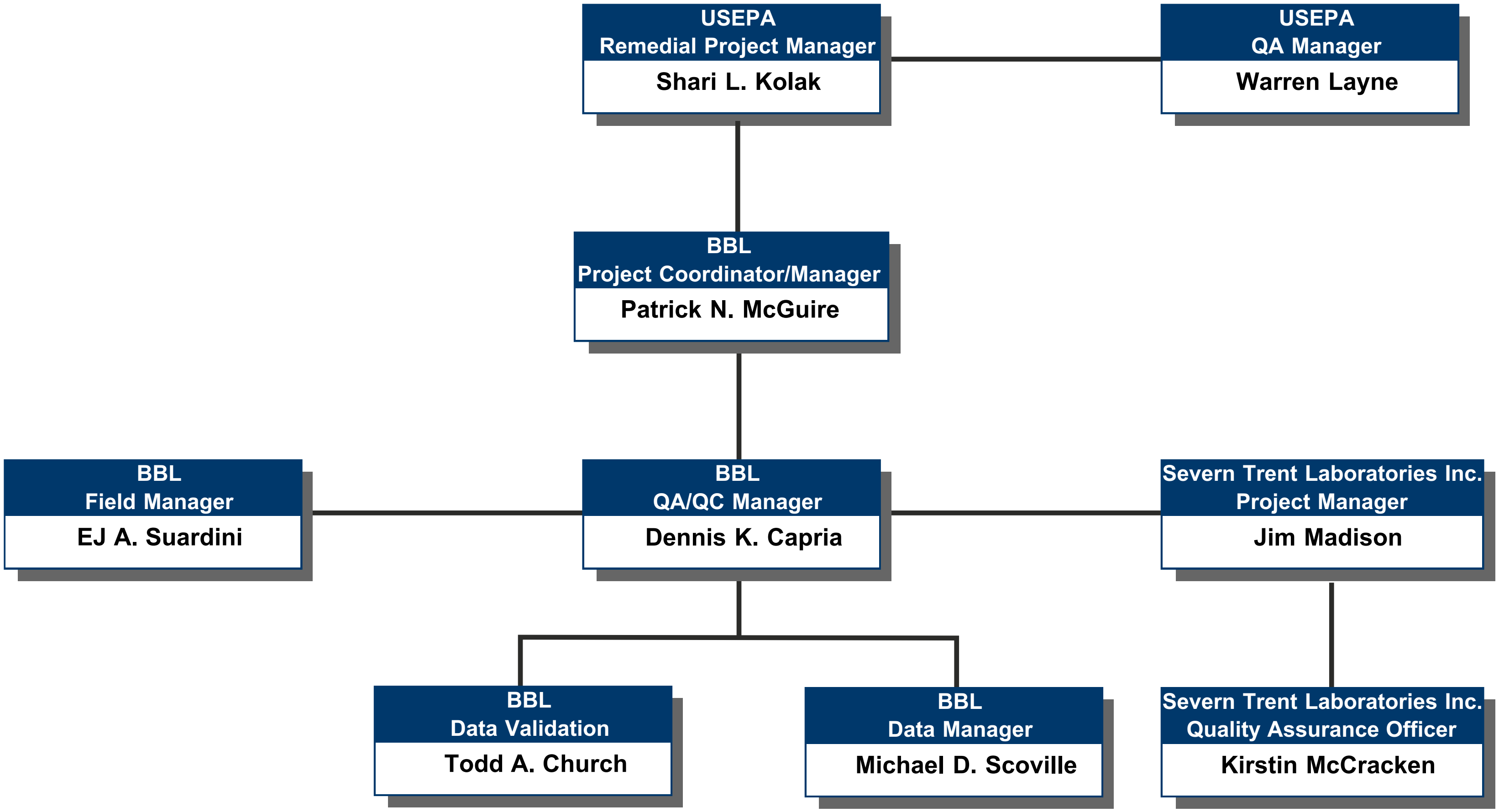
64585X00 66090X03.TIF



GEORGIA PACIFIC CORPORATION
KALAMAZOO MILLARD FORMER HAWTHORN MILL
TIME CRITICAL REMOVAL ACTION

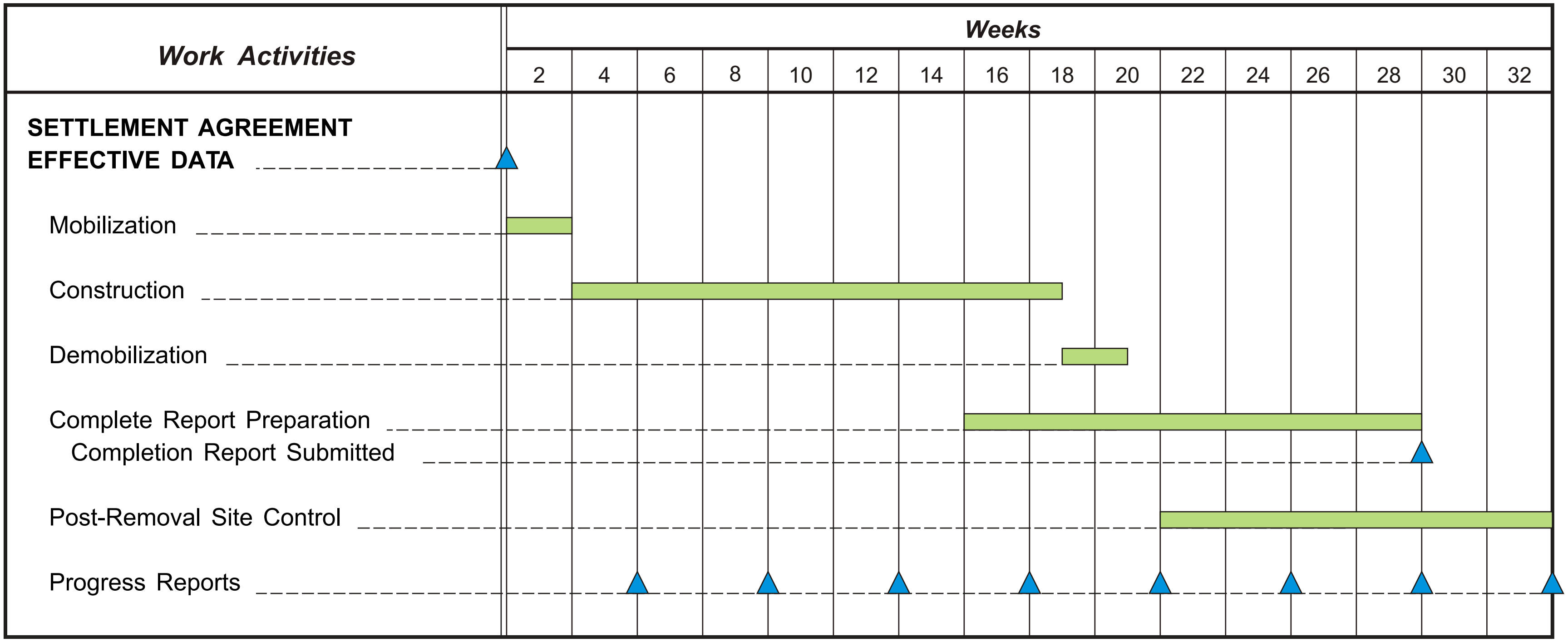
QAPP ORGANIZATION CHART

BBL® an ARCADIS company

FIGURE 2

8/03/06 SYR-85-KLS
99090aaa/99090F03.CDR

DRAFT



NOTES:

1. Approval to proceed includes approval of TCRA Workplan and associated documents
2. Construction includes removal of material from refuse and oxbow area, transformer pad, wastewater pipeline, and restoration of each area.

GEORGIA-PACIFIC CORPORATION
KALAMAZOO MILL AND FORMER HAWTHORNE MILL
TIME CRITICAL REMOVAL ACTION

PROJECT SCHEDULE

BBL®
BLASLAND, BOUCK & LEE, INC.
engineers, scientists, economists

FIGURE
3

05/15/06 SYR-D85-DJH
64585675/64585j01.cdr

Figure

Appendices

1. Project Management and Objectives

This *Quality Assurance Project Plan Addendum* (QAPP Addendum) updates the QAPP (Blasland & Bouck Engineers, P.C. [BBEPC], 1993a) developed to support the *Remedial Investigation/Feasibility Study Work Plan* (RI/FS Work Plan) for the Allied Paper Portage Creek Kalamazoo River Superfund Site (BBEPC, 1993b).

This QAPP Addendum specifically identifies the protocols and methods which will be employed to assure the quality of data collected as part of the Time-Critical Removal Action (TCRA) for the removal of paper-making residuals (residuals) and soils that contain, or may contain, polychlorinated biphenyls (PCB) from the Georgia-Pacific Corporation (Georgia-Pacific) Kalamazoo Mill Property (Kalamazoo Mill Property) and the former Hawthorne Mill Property (Hawthorne Mill Property), collectively referred to as the Mill Properties (Figure 1).

The specific sampling requirements and the locations and numbers of samples to be taken, are found in the *TCRA Work Plan* (Work Plan) (BBL, 2006). The Work Plan provides the rationale for the locations and numbers of samples and the selection of measurements and chemical analytes.

The procedures specified herein will be used for the sampling and analysis of soils, residuals, ambient air, and water for PCB to determine if specified performance standard are met or action levels are exceeded. Additionally post excavation soil samples will be analyzed for other constituents to characterize the soil. Turbidity in surface water will be monitored in the Kalamazoo River at locations upstream and downstream of the excavation activities in the Refuse Area. In addition, dust generation will be monitored during TCRA construction activities that potentially may generate dust.

1.1 Project Organization

BBL maintains overall technical responsibility for the TCRA at the Mill Properties. As such, BBL will perform sampling associated with construction activities, compile and report resulting data, provide quality assurance/quality control (QA/QC) oversight, and prepare all associated reports.

The direct management of the technical and administrative aspects of the TCRA will be accomplished by representatives of Georgia-Pacific, BBL, and United States Environmental Protection Agency (USEPA) Region 5. Currently, the following personnel have been assigned to this project:

Affiliation	Title	Name	Phone #
USEPA Region 5	Remedial Project Manager	Shari L. Kolak	312-886-6151
USEPA Region 5	Quality Assurance Manager	Warren Layne	312-886-7336
BBL	Project Coordinator/Manager	Patrick N. McGuire	315-671-9233
BBL	Field Manager	EJ Suardini	810-229-8594
BBL	Quality Assurance Manager	Dennis K. Capria	315-671-9299
BBL	Data Manager	Michael D. Scoville	315-671-9387

The analytical laboratory services for this project will be provided by Severn Trent Laboratories, Inc. (STL) in Burlington, Vermont. STL is accredited under the National Environmental Laboratory Accreditation Program (NELAP). A certificate of accreditation is provided in Appendix A.

Affiliation	Title	Name	Email address	Telephone #
Severn Trent Laboratories, Inc.	Laboratory Project Manager	James Madison	jmadison@stl-inc.com	802-655-1203
	Quality Assurance Officer	Kirstin McCracken	kmccracken@stl-inc.com	802-655-1203

Figure 2 presents the organization chart for this TCRA QAPP Addendum.

1.2 Project Description

1.2.1 Project Overview

The scope of work for the TCRA at the Mill Properties is detailed in the Work Plan and consists of the following activities:

- Excavate residuals and soils that contain, or may potentially contain PCB concentrations exceeding performance standard of 10 mg/kg from the Refuse Area and Oxbow Area and dispose of them at the A-Site (Figure 1).
- Excavate visibly stained soil from beneath the Transformer Pad Area and dispose of it at a Type II landfill.
- Sample soil after excavation and characterize it for Target Compound/Target Analyte List (TCL/TAL) constituents.
- Excavate the pipeline and wet well at the Wastewater Pipeline Area and dispose of them at the A-Site.
- Restore the Refuse Area and Oxbow Area.

1.2.2 Project Schedule

A tentative schedule for the Mill Properties TCRA is shown on Figure 3 in the TCRA Work Plan and included in this QAPP Addendum. The schedule will be updated as necessary and reported in the monthly reports prepared for the TCRA.

1.3 Project Planning and Problem Definition

1.3.1 Project Planning Meetings

Project planning meetings and/or teleconferences have been and will continue to be scheduled as needed to develop the TCRA and monitor ongoing work activities detailed in the Work Plan. Meetings involving Georgia-Pacific and USEPA Region 5 will be coordinated through the Project Coordinator or designated representative.

the removal activities, an action level of 0.2 μg/m^3 for the third location shown on Figure 1 will be used, which will be positioned near the work area. If an action level is exceeded, the USEPA will be notified and corrective actions will be taken to reduce emissions. It should be noted, as conditions change or removal activities move to new locations the air samplers may move to new locations, as well. Any new air sampler location will be selected after consultation with USEPA On Scene Coordinator.

The air monitoring program will follow the procedures outlined by USEPA Method TO-4A from the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air* (USEPA, 1999) for sample collection and analysis. Sampling will be conducted daily for 5 days during commencement of remediation activities at the Mill Properties. Samples will be collected during the entire work day. If the first week's data demonstrate that concentrations at the monitoring locations are below the action levels and similar activities are planned for subsequent weeks, the frequency of sampling may be reduced or terminated upon approval by the USEPA. Following a reduction in sampling frequency, if the nature of the work changes significantly, air monitoring may be resumed.

Meteorological data will be recorded during sampling days. Approximate wind direction, wind speed, and general weather conditions will be obtained from the Battle Creek/Kalamazoo International Airport.

Turbidity monitoring will be performed in the Kalamazoo River approximately 100 feet upstream and 100 feet downstream of excavation activities in the Refuse Area during periods of active work. Measurements of turbidity at the mid-depth point of the water column will be recorded daily (2 hours into the start of the work day). Turbidity monitoring will be conducted consistent with the Remedial Action Turbidity Monitoring Plan (TMP) (BBL, 1999).

At the request of USEPA a relationship between turbidity and TSS has been developed. This relationship is provided in Equation 1 below:

$$TSS = O.5016T^{1.4} \quad (l)$$

where: TSS = Total Suspended Solids
T = Turbidity

Equation 1 was developed using Kalamazoo River turbidity and TSS data collected during the Kalamazoo River Supplemental Investigation 2000-2001.

If excavation activities progress to within close proximity of the Oxbow Area channel, turbidity monitoring may also be performed at appropriate upstream and downstream locations in the oxbow channel, if necessary.

Water collected from temporary staging/dewatering areas, decontamination fluids, and other liquids generated during construction activities will be treated onsite at a temporary water treatment system (TWTS) located on the South side of the Area East of Davis Creek (Figure 1). The TWTS will consist of filtration and liquid-phase granular two-stage activated carbon. The two-stage activated carbon treatment system will be used so that rotation and replacement of the carbon tanks will occur immediately upon detection of PCB at the intermediate stage. Water will be collected, handled, treated, monitored, and discharged to Davis Creek. To monitor the TWTS, an influent, intermediate (i.e., between the carbon stages), and effluent wastewater sample will be collected and analyzed for PCBs and total suspended solids (TSS) from the TWTS prior to any discharge of the treated water. Treated wastewater will be stored in 20,000 gallon frac tanks until sampling and analysis confirm that the discharge limitations (i.e., 2.6×10^{-5} μg/L for PCBs [or not detected] and 45 mg/L for TSS) have been achieved prior to discharging the water to Davis Creek. Sampling procedures, preservation and handling, and analytical protocol for monitoring for PCB will be consistent with USEPA Method 608 (the quantification level

2.5 Laboratory Analytical Method Requirements

The maintenance and calibration requirements for the standard fixed laboratory instruments used to perform these analyses are specified in the laboratory-specific SOPs are listed in Table 1-4 and included as Appendix B.

2.5.1 Laboratory Information

Laboratory QA Plans are maintained at the laboratory facilities. See Section 1.1 for laboratory key project personnel and contact information.

2.6 Quality Control Requirements

2.6.1 Field Sampling and Analytical Quality Control

Field sampling QC requirements are summarized in Tables 1-5 and 2-1 which defines the collection frequency and acceptance criteria for the following field QC samples:

- field equipment rinseate blanks;
- field duplicates; and
- sample preservation requirements.

2.6.2. Laboratory Analytical Quality Control

Laboratory analytical QC requirements are described in detail in the published methods (e.g., SW-846). Laboratory SOPs and project-specific requirements are documented in Tables 1-1A thru C and 1-4. If a difference is noted in QC specifications included in the USEPA methods, laboratory SOPs, or Tables 1-1 thru 1-8 of the 1993 QAPP, the Analytical Laboratory Quality Control Checks specified in this 2006 QAPP Addendum take precedence and will be used to evaluate the validity and usability of the data generated during the verification sampling (see example in table below).

Parameter	Accuracy - Percent Recovery		Precision - RPD	
	MS/MSD 1993 QAPP	MS/MSD 2006 QAPP Addendum	MS/MSD 1993 QAPP	MS/MSD 2006 QAPP Addendum
PCB (Soil)	29 – 131	29 - 150	50	30

MS/MSD = Matrix spike/matrix spike duplicate
RPD = Relative Percent Difference

Performance and system audits will be completed during this project to maintain high quality data. These audits are described in Section 10.2 of the 1993 QAPP. Preventative maintenance procedures are described in Section 11.2 of the 1993 QAPP.

2.7 Data Reduction, Validation, and Reporting

Data reduction, validation, and reporting procedures will be consistent with Section 8 of the 1993 QAPP, with the exception of the following data validation guidance referenced below:

Analysis	Guidance Documents
Organics	*National Functional Guidelines for Organic Data Review* (USEPA, 1999b).
Inorganics	*National Functional Guidelines for Inorganic Data Review* (USEPA 2002).

Data quality indicators are discussed in Section 12 of the 1993 QAPP.

2.8 Corrective Action

Corrective Action procedures are followed to maintain data quality. Corrective actions are discussed in Section 13 of the 1993 QAPP.